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RESEARCH, TEST AND
EVALUATION PERFORMED AT
NAVAL CONSTRUCTION
BATTALION CENTER,
GULFPORT, MS, FOR THE USAF
INSTALLATION/RESTORATION
PROGRAM

R.W. HELSEL, R.W. THOMAS

EG&G IDAHO P.O. BOX 1625 IDAHO FALLS ID 83415

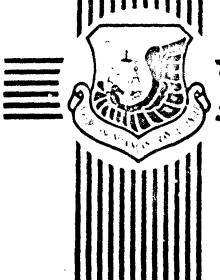
**JANUARY 1988** 

FINAL REPORT

JUNE 1985 - JULY 1985

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AIR FORCE ENGINEERING AND SERVICES CENTER
TYNDALL AIR FORCE BASE, FLORIDA 32403

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capability to treat dioxin-contaminated soil and may be considered for full-scale coil rectoration at USAF HC sites. Sensitivity analyses of six variables (geographic location, soil quantity, electrical power prices, labor, capital equipment use charge and transportation) were performed to estimate cost for conditions other than those found at NCLC. The process may nave application for treatment of more easily pyrolyzed organic compounds such as semivolatiles, pesticides, and polychlorinated biphenyls, as well as some inorganics.

This report is organized into two volumes: Volume I presents the final report on the performance of an advanced electric reactor for use in decontaminating soil containing Herbicide Orange. Volume II presents supplementary analytical data and historical information that supports the the research findings reported in the first volume.



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#### VOLUME II

#### SECTION I INTRODUCTION

#### A. OBJECTIVE

The objective of this program is to demonstrate the feasibility of using a thermal-pyrolysis technology for soil cleanup and restoration of a Herbicide Orange (HO)-contaminated site at the Naval Construction Battalion Center (NCBC) at Gulfport, Mississippi. This program is under the sponsorship of the Air Force Engineering and Services Center (HQ AFESC), Tyndall Air Force Base, Florida. The objective is twofold:

- 1. Perform a field demonstration with a pilot-scale unit at the NCBC location using the Advanced Electric Reactor (AER) process owned by the J. M. Huber Corporation of Borger, Texas. The AER is a high-temperature fluid wall reactor.
- 2. Provide technical evaluation and cost estimates for full-scale cleanup/site restoration using the AER technology, which would provide information to compare this technology with others.

A specific goal of this technology testing was to reduce the total isomers of tetra, penta, and hexachlorodibenzo-p-dioxin and respective isomers of polychlorodibenzofuran to less than 1 part per billion (ppb). The overall soil treatment goal of the demonstration was to reduce the level of contaminants to criteria acceptable to Headquarters, U.S. Environmental Protection Agency (EPA) to facilitate the delisting of the soil under the auspices of the Resource Conservation and Recovery Act (RCkA) of 1976, as amended by the Hazardous and Solids Waste Amendments (HSWA) of 1984.

The AER field demonstration was one of two technologies selected for the Air Force Small-Scale Demonstration Program. Those technologies are being evaluated for decontamination treatment of former Department of Defense (DOD) HO sites. The purpose of the research demonstrations is to provide actual field data on the feasibility of the technology so that scaleup and cost-effectiveness can be determined for future restoration efforts. The other small-scale technology undergoing research is a thermal desorption process being performed by the IT Corporation (ITC) at NCBC and Johnston Island (Pacific Ocean). Results of these tests appear in separate reports.

#### B. BACKGROUND

NCBC is a fenced, limited-access military installation. It is a land area of several square miles located approximately 2 miles from the Gulf of Mexico, and is approximately 20 feet above sea level.

Approximately 12 acres at NCBC served as an HO storage site. The storage site was stabilized with Portland cement approximately 30 years ago. The stabilized soil provided a hardened storage area for heavy supplies and equipment. Over the years, additional fill materials (shell, rock, soil, asphalt, and tar) were added to the storage area, providing a cover of up to several inches over the cement-stabilized soil. Through use, the contaminated site is now about 18 acres. During 1980, retention basins were constructed on the storage site to prevent migration of dioxin-contaminated soils offsite by surface runoff. Currently, the storage site within the fenced perimeter is a restricted area and is not used.

#### C. SCOPE

Volume I of this report presents the results of a pilot-scale pyrolysis process to treat NCBC soil contaminated by polychlorodibenzo-polioxins and polychlorodibenzofurans and presents a cost estimate for full-scale remedial action by this process. This volume presents appendices to document supporting data.

#### APPENDIX A

# REQUEST FOR AND EPA AUTHORIZATION LETTERS FOR AIR FORCE ENVIRONMENTAL RESTORATION TECHNOLOGY, RESEARCH, AND TEST EVALUATION PROGRAM AT NCBC

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P.O. BOX 1625, IDAHO FALLS, IDAHO 83415

bcc: K. L. Falconer F. C. Fogarty
T. H. Smith

D. L. Uhl Central Files H. D. Filliams File

March 14, 1985

Mr. Paul des Rosier
Deputy Chairman, Dioxin Disposal Advisory Group
Environmental Protection Agency
401 M Street SW
Washington, DC 20460

TRANSMITTAL OF TECHNICAL INFORMATION FOR USAF RESEARCH AND TEST EVALUATION -HDW-4-85

Dear Mr. des Rosier:

Based on previous discussions with the Dioxin Disposal Advisory Group (DDAG) and following the guidance provided by DDAG, EG&G Idano, Inc. has prepared the attached document for review by DDAG. The document presents technical information concerning the Research Test and Evaluation activities of the United States Air Force (USAF) Environmental Restoration Program for former Herbicide Orange storage sites.

Captain T. L. Stoddart, USAF, Engineering Services Center, (HQ AFESC) has arranged for a presentation of this information to the DDAG in Washington, DC, on March 21, 1985 at 1000 hours. Representatives of EG&G Idaho, Inc. and its subcontractors, the IT Corporation and J.M. Huber Company, will be present to provide additional information or answer questions as they arise. Enclosed are eleven copies of the document for you to distribute at your discretion.

On behalf of the USAF Engineering Services Center and our subcontractors, we are pleased to present this information to you and will look forward to further discussions on March 21, 1985. If questions arise prior to that date, please contact me at FTS 583-1763 or K. L. Falconer at FTS 583-1559.

Very truly yours,

H. D. Williams

Senior Program Specialist Hazardous Waste Program March 14, 1985 Mr. Paul des Rosier HDW4-85 Page 2

#### ag

Enclosure: as Stated

cc: I. Aoki, DOE-ID M. Cook, EPA

K. Kleveno, EPA J. McGraw, EPA

T. L. Stoddart, Captain, USAF

J. O. Zane, EG&G Idaho (w/o Enclosure)



## Appendix A, Exhibit 2 UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460

AFR 25 1985

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

Colonel Rebert Boyer
HQ AFESC/RD
Tyndall Air Force Base, FL 3240

Dear Colonel Boyer:

We have reviewed your document entitled "Environmental Restoration Technology -- Research and Test Evaluation, " informing the Environmental Protection Agency (EPA) of your intent to treat soils contaminated with 2,3,7,8-tetrachlorodibenzo-p-dicxin (2,3,7,8-TCDD). We understand that you wish to conduct a series of research tests on less than 3,700 pounds of soil (less than two tons, or two cubic yards) contaminated with approximately 200 ppb of 2,3,7,8-TCDD. The 2,3,7,8-TCDD-contaminated soil is located at the Naval Construction Battalion Center (NCBC), Gulfport, MS, the site of the research tests. We also understand that you plan to destroy the 2,3,7,8-TCDD in the soil by testing two treatment units for approximately three to five weeks. The two units are: 1) thermal pyrolysis using the Advanced Electric Reactor developed by the J. M. Huber Company, and 2) thermal desorption followed by ultraviolet light destruction, developed by the IT Corporation. Recause the destruction tests are being conducted for research purposes, you have requested a waiver from notification under 40 CFR Part 775.

On March 21, 1985, the Dioxin Disposal Advisory Group (DDAG) met with the U.C. Air Force to discuss the details of the planned research and evaluation studies. As a result, the DDAG determined that the proposal involves potentially feasible technologies, that the technical, safety, and environmental factors have been adequately addressed, and that the research activities will provide useful information in the destruction of 2,3,7,8-TCDD-contaminated soils. Thus, a research waiver from the notification requirements under 40 CFR Part 775 is hereby granted to the U.S. Air Force (HO AFESC) to conduct the research tests. The waiver is being granted since the quantity of soil is small, the equipment being used for the research is pilot scale, and the tests to be conducted are of short duration. If testing should continue beyond July 15, 1985, the effective date of the RCRA dioxin regulation (50 FR 1978-2006; January 14, 1985), the activities will be subject to the provisions of that rule.

Your research at Johnston Island, however, will occur after July 15, 1985. As such, it will be subject to the RCRA dioxin listing. As discussed with members of your staff, EPA is proceeding with the preparation of a research development and demonstration permit. If you have any questions, please feel free to contact Dr. Howard Fribush, Office of Solid Waste, on (202) 475-6678.

Sincerely yours,

Jack W. McGraw Acting Assistant Administrator

cc: Captain Terry Stoddart
CQ AFESC/RDVW
Tyndall Air Force Base

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#### APPENDIX B

### REQUEST FOR AND EPA AUTHORIZATION LETTERS FOR DIOXIN-CONTAMINATED WASTE DISPOSAL

			Page
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Exhibit	2.	EPA Authorization Letter	14

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#### DEPARTMENT OF THE AIR FORCE

HEADQUARTERS AIR FORCE ENGINEFRING AND BERVICES CENTER
TYNOALL AIR FORCE BASE, Pt. 32403

24 June 85

Reply to attn of: RDVW

subj: Herbicide Orange Waste Disposal; Letter of Transmittal

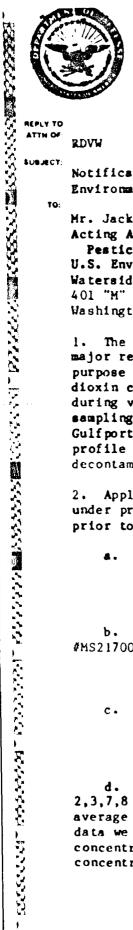
T0: Commanding Officer
Naval Construction Battalion Center
Gulfport, MS 39501
code 470

- 1. Please find attached copies of documents pertaining to the disposal of Herbicide Orange contaminated wastes that resulted from HQ AFESC/RDVW research projects at the Naval Construction Battalion Center(NCBC). Tab #1 is the TOSCA notification submitted to the Environmental Protection Agency. Tab #2 is the Environmental Protection Agency letter authorizing the Air Force to dispose of the described waste.
- 2. The exact number of drums scheduled for disposal will be determined on the last day of packing. The total number of drums will be less than that stated in the permit application. The reduced number of drums resulted from waste compaction that reduced the total volume to be disposed. The description of the waste is accurate, the only variation is that the soltrol solvent described will be shipped as a solidified material, the solidification step was a requirement stipulated by Rollins inc.
- 3. The generation of the described waste material is a one time only action related to our research at NCBC. No additional disposal requirement is projected.
- 4. Should you have questions please contact me at (601)-864-0056.

Terry L Stoddart, Capt, USAF, BSC

Project Manager

attch
tab #1 TOSCA letter
tab #2 EPA authorization



#### DEPARTMENT OF THE AIR FORCE

HEADQUARTERS IN FORCE ENGINEERING AND SPRYICES CENTER TYMDALL AIR FORCE BASE, FL 22403

APR 25 1985

REPLY TO ATTH OF

SUBJECT:

RDVW

Notification of Disposal of TCDD (Dioxin) Contaminated Waste from Air Force Environmental Restoration Activities

Mr. Jack McGraw Acting Assistant Administrator for Pesticides and Toxic Substances U.S. Environmental Protection Agency Waterside Mall 401 "M" Street, S.W. Washington D.C. 20460

- 1. The U.S. Air Force Installation Restoration Program is involved with two major research activities at former Herbicide Orange storage sites. The purpose of this application/notification is to provide for the disposal of dioxin contaminated personnel protection and sampling equipment generated during various phases of our two programs. Presently, surface and subsurface sampling is being conducted at the Naval Construction Battalion Center (NCBC), Gulfport MS and Eglin AFB (EAFB), Fort Walton Beach FL, to determine the profile and extent of contamination. Follow on phases involve testing soil decontamination technologies at NCBC.
- 2. Application for approval is made for disposal of TCDD contaminated waste under provisions of 40CFR, Part 775, 190(b). Disposal will be accomplished prior to 15 Jul 85.
  - HQ Air Force Engineering & Services Center Name and address of firm: Engineering and Services Laboratory (HQ AFESC/RD) Tyndall AFB FL 32403 6001 ID No. FL 1570024124
- b. Site 1: Naval Construction Battalion Center, Gulfport MS, ID #MS2170022626.
  - Site 2: Eglin AFB, Fort Walton Beach FL, ID# FL572024366.
  - c. Point of Contact: Capt Terry L. Stoddart HQ AFESC/RDVW Tyndall AFB FL 32403 (904) 283-2942
- d. A review of current analytical data indicates the maximum levels of 2,3,7,8 TCDD contamination in soils from NCBC and EAFB is 300 ppb. The average concentration in these soils ranges from 20-30 ppb. Based on these data we anticipate that the drummed waste will contain substantially lower concentrations of 2,3,7,8 TCDD. The soil is also contaminated with varying concentrations of 2,4,D and 2.4,5T, ID Nos. D016 and D077, respectively.

- d. Quantity of Waste: The first phase of soil sampling at NCBC resulted in the generation of 27 drums of contaminated clothing. The subsurface sampling scheduled for NCBC in early May will generate another 22 drums. Similar activities at EAFB are anticipated to generate 22 drums of contaminated clothing. The follow on phase of the project, which involves testing of soil decontamination technologies, scheduled for Jun 85, will generate approximately 54 drums of contaminated clothing. Currently, no technology demonstrations are scheduled for Eglin AFB FL. One of the technologies scheduled for demonstration at NCBC will produce 75 gallons of dioxin-contaminated solvent. The solvent, Solitrol<sup>®</sup>, is a petroleum product manufactured by Phillips. The solvent has a flashpoint of 185°F, pH 7, and a copper strip corrosion of 1.0. It is anticipated that the dioxin contamination in the solvent will be less than 100 ppb. A total of 127 drums are scheduled for disposal. The waste to be disposed consists of 125 drums of contaminated chemical protective equipment and two drums of contaminated solvents.
- e. All waste will be packaged, labeled, and transported in accordance with existing EPA, DOT, and state regulations. Wastes will be disposed by incineration at Rollins Environmental Services, Inc., Deer Park TX, EPA ID No. TXD0551141378.
- f. Status of Waste: The 27 fiber drums of contaminated wastes are stored in a open-sided metal storage shed at NCBC. The shed has a concrete floor. It is surrounded by a 6 foot high chain link fence topped with barbed wire. The drums are stacked one high on wood pallets and covered with 6-mil plastic sheeting. This storage facility is located inside the contaminated area, which is surrounded by a fence and posted as a restricted area. Waste presently generated will be stored in a similar manner until pickup for transportation to Rollins Environmental Services, Inc., which is scheduled for the last week of Jun 85.
- 3. Your time and effort for consideration of this approval request is appreciated. If questions should arise, please contact Capt Terry Stoddart, Headquarters Air Force Engineering and Services Center, Engineering and Services Laboratory (HQ AFESC/RDV), Tyndall AFB FL 32403-6001; (904) 283-2942.

James R. Van Orman

JAMES E VAN ORKAN
Doputy Director of
Engineering & Services Leboretery

cc: EG&G Idaho (Mr. Williams)
U.S. EPA, Region 4
Mr. DesRosien, EPA/ORD
Mr. Kleveno, EPA/HRSD
Mr. Cummins, EPA/OSWER
325CES/DEEV
AD/DEV

NCBC/Code 470

Int cc AFESC/DEV

WH-562B/H. Fribush/ht/S242K/475-6726/05-23-85/02/ht/26

MAY 3 1 1985

Mr. James R. Van Orman
Deputy Director of
Engineering & Services Laboratory
Department of the Air Porce
Deadquarters Air Force Engineering and Services Center
Tyndall Air Force Base, FL 32403

Dear Mr. Van Orman:

SOCIALIA SESECETA ESCLUSIOS ESPERINAS CONTRACAS DE CONTRACAS DE CONTRACAS DE LA CONTRACAS DE LA CONTRACAS DE CONTRACAS. DE CONTRACAS DE

We have reviewed your letter of April 25, 1985, informing the Environmental Protection Agency (EPA) of your intent to dispose of waste materials contaminated with 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). We understand that you have about 125 drums of 2,3,7,8-TCDD-contaminated clothing and two drums of 2,3,7,8-TCDD-contaminated solvent. We also understand that you wish to dispose of this waste by incineration at Rollins Environmental Services, Deer Park, Texas.

The Agency's Dioxin Disposal Advisory Group (DDAG) has no objections to your planned disposal. We recommend that the incinerator be operated under the conditions that have demonstrated a destruction and removal efficiency (DRE) of 99.9999 percent for PCBs.

If you are unable to proceed with the planned disposal or if you choose an alternative method, please be advised that you are required to submit a new notification prior to disposing of 2,3,7,8-TCDD-contaminated waste materials. It should be noted that, after July 15, 1985, the effective date of the RCRA listing regulation (50 FR 1978-2006; January 14, 1985), which designates certain 2,3,7,8-TCDD-contaminated wastes as hazardous, you will be subject to the provisions of that rule. If you have any questions, please feel tree to contact Dr. Howard Fribush, Office of Solid Waste, on (202) 475-6726.

Sincerely,

Miphin R. Lanusy

Mack W. McGraw

Acting Assistant Administrator

#### APPENDIX C

### PUBLIC NOTIFICATION AND LOCAL NEWS ARTICLES ON TECHNOLOGY, RESEARCH, AND TEST EVALUATION PROGRAM AT NCBC

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Exhibit	1.	The Daily Herald, Friday May 24, 1985	17
Exhibit	2.	The Sun, May 25, 1985	18
Exhibit	3.	The Daily Herald, May 29, 1985	19
Exhibit	4.	The Sun, June 6, 1985	20

The Daily Herald FRI. May 24, 1985

> PUBLIC NOTIFICATION Notice is hereby given of the public availability of a Toxic Substance Control Act (TOSCA) document titled "Environmental Restoration Techniogies: Re-search Test and Evaluation" cavering work to be conducted by the United States Air Force, at the Naval Construction Battallon Center, Gulfport, Mississippi. The document, available for review at the Guifport-Harrison County, Library, 21st Avenue, Guifport, Alssissippi, covers the scope, procedures, background and goals of research to be conducted by the Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndait AFB, Fiorida. The research is aimed at discovering the most efficient, cost-effective method(s) of removing environmental contaminants from soil. For questions beyond the scope of this document, please contact the Directorate of Public Affairs, Ale Force Engineering and Services Center, Tyndall AFB, Florids 32403, Telephone: (904) 283-464 V-76, adv. 23, 41.

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Center, Eglin Alr Force Base,

the cleanup of three dloxin-con Air Force will devise a plan for million research and develop

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The testing is part of a \$1.7

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to 200 to 300 parts per billion. detected in concentrations of up Seabee Center, dioxin has been According to the Centers for Dis-In soil samples taken at the this year or early next year prohably will not begin until late the sites suitable for normal use, The cleanup, designed to mak

502

### Permits for soil tests not needed

By TOM CHARLIER STAFF WRITER

The U.S. Air Force will not need Mississippi environmental permits to carry out testing next week on dioxin-contaminated soil at the Gulfport Seabee Center, state officials said.

The Pollution Control Permit Board, which was briefed Tuesday on work to be done at the base, will not require the Air Force, or its two contractors in the work, to undergo the normal permitting procedure, said Jack McMillan, who heads the Bureau of Pollution Control's solid-waste section.

The two companies selected to do the testing - J.M. Huber Co. of Atlanta, and the IT Corp. of Washington, D.C. have begun delivering equipment to the base in preparation for the work, which is scheduled to take place June 5. The contractors will test experimental methods involving the use of heat and chemicals to remove dioxin from soil.

"Right now, we don't see any reason for issuing a permit, because it's just such a minute amount (of dioxin) they'll be dealing with," McMillan said.

The state, however, will require permits before any full-scale cleanup effort begins at the site, he added.

8gt. Jim Denny, a spokesman for the Engineering and Services Laboratory at Tyndall Air Force Base, Fla., said the Air Force has provided the Bureau of Pollution Control with complete details of the work, which he said will not

See AGENT, Page A-2

### Daily Herald 5/29/85

## Air Force's dioxin tests won't require state permits

By TOM CHARLIER Staff Writer

The U.S. Air Force will not need Mississippi environmental permits to carry out testing next week on dioxin-contaminated soil at the Gulfport Seabee Center, state officials said.

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Sgt. Jim Denny, a spokesma for the Engineering and Service Laboratory at Tyndall Air Fore Base, Fla., said the Air Fore has provided the Bureau of Pelution Control with complete d tails of the work.

The soil to be tested is on a sealed-off 12-acre portion of the base where thousands of drums of Agent Orange, a herbicide used to defoliate jungles during the Vietnam War, were stored from 1968 to 1977. The herbicide contained dioxin, a contaminant produced during its manufacture, which remains in soil at the site as a result of leaks.

Dioxin has been shown to be extremely toxic in laboratory studies, with doses of as little as 5 parts per trillion producing cancerous tumors in test animals, according to some researchers. But the substance's effects on humans are not fully understood.

In soil samples taken at the Seabee Center, dioxin has been detected in concentrations of up to 200 to 300 parts per billion. According to the Centers tor Disease Control in Atlanta, a concentration of 1 part per billion in soil is sufficient to warrant concern.

The Come coper 185

### Workers begin decontaminating dioxin-tainted soil at Seabee 19 ns

By TOM CHARLIER

Technicians on Warts pgen processing distinguished end at the passes distributed in Guifport in ora that could lead to an eventual cleanup of a site where the herbicide Agent Orange was stored.

Specially-suited workers from IT Corp. of Knuzville. Tenn., were scheduled late last night to begin treating soil by an untested device designed separate and destroy dicate Shrough the use of heat, shrough and uttravious batts. The well had been rescheduled from afternoon to nighttime because of the heat.

Later this month, crefrom another firm, J.M. Hubes Co. of Borger, Texas, are slated to demonstrate an experimental process using a device known as an advanced electrical reactor. That process destroys dioxin with extremely high temperatures.

Both firms are under contract with the U.S. Air Force to process a total of 3,500 pounds of contaminated offi at the base. The work is roll of arresearch offers. directed by the Air Porce, the U.S. Environmental ryflection Agency and the U.S. Department of Energy to identify a "useful technolto treat contaminated soil at three military installations the Guifport base, Eglin Air Force Sase, Fla , and Johnston Island, in the Western Pacific Ocean

With the tests, "we II be able to determine whether or not it will be economically feasible to clean up the site," Air Force Maj. Jim Heaberg said.

It now costs between \$500 and \$1,000 a cubic yard to dispose of contaminated soil at Memphout the country.

Membranianing the soil may
below a less-expensive afternative, the Air Force efficials said. SUN

ABOUT DEFAUT OF THE PART million earmarked for the research effort - which includes soil sampling and other monitoring airwady done at the three bases — will be spent in Guifport. The higher costs are attributed to the costs are attributed to the problems posed by the unique soil ownlitions there, officials said.

We're patitud wir money against the most difficult problem," said Wayne R. Mathis, an EPA engineer.

Heatery said the zurtace o the storage atte is composed of a Trixture of asphalt, see. gravel and crushed shell.

Agent Orange, used to defoliate jungies during the Vietnam War, was stored at a 12-acre site on the Seabee Center between 1965 and 1977. The more than \$40,000 gallons kept at the base in later years were among the 2.4 million gallons of the herbicide destroyed on a wante-burning meto in the Parties Town

Dienia, a byproduct of the sandacture of Agent Orange, extremely toxic in tests on leboratory animals. Soil samples at the former storage area have revealed dioxia concentrations of as high as 200 parts per billion bove widely established safe levels

The storage area is scaled off, and state and federal officials contend no significa contamination has been found

The testing at the Seaber The resting at the neanee Center must be completed by July 18. EPA and the Mississippi Burean of Polistien Control have maked the usual hazardous waste permit requirements for the testing until that date.

6-6-85

#### APPENDIX D

TEST PLAN FOR HUBER'S AER DEMONSTRATION TEST AT NCBC

TEST PLAN TO DEMONSTRATE HUBER'S ADVANCED ELECTRIC REACTOR ON DIOXIN CONTAMINATED SOILS AT GULFPORT, MISSISSIPPI



### **HUBER TECHNOLOGY**

J. M. HUBER CORPORATION

P. C. Box 2831 Borger, Texas 79008-2831 (806) 274-6331 TEST PLAN TO DEMONSTRATE HUBER'S ADVANCED ELECTRIC REACTOR ON DIOXIN CONTAMINATED SOILS AT GULFPORT, MISSISSIPPI

Submitted To:

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J. M. Huber Corporation
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Borger, Texas 79007

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#### 1. INTRODUCTION

This test plan provides a detailed description of Huber's plans to conduct a test for the research and test evaluation of the Advanced Electric Reactor (AER) to treat soil contaminated with Herbicide Orange at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi. This test will be conducted under contract to EG&G Idaho, Inc., Idaho Falls, Idaho.

Huber will transport its mobile 3" AER to Gulfport, Mississippi, and process approximately 1,000 pounds of soil containing the toxic 2,3,7,8 tetrachlorodibenzo-p-dioxin (TCDD) at NCBC. The actual test will require about 30 to 40 hours of operation and be performed on June 24 and 25, 1985.

In September 1983, Huber embarked on an aggressive program to demonstrate the capability of the AER to decontaminate soil. This program includes the treatment of soils contaminated with HCB, PCB, carbon tetrachloride, octochlorodibenzo-p-dioxin, and TCDD. The Gulfport test represents a continuation of this demonstration process.

All of the tests conducted to date have been successful in destroying the contaminate being tested while protecting the environment.

These results clearly demonstrate the extremely high destruction capabilities of Huber's AER process and its intrinsic safety advantages over conventional treatment methods.

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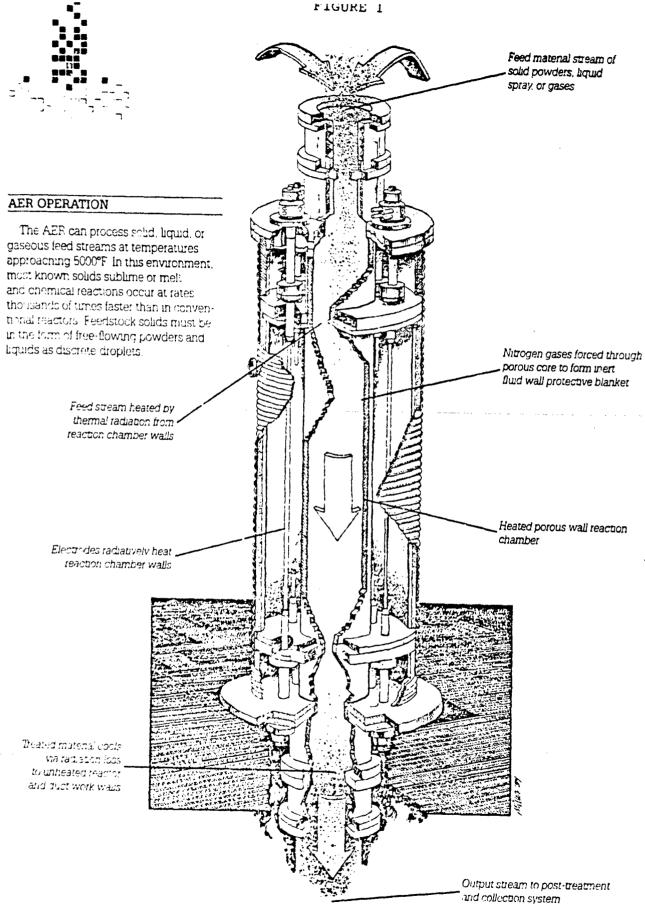
#### 2. PROCESS DESCRIPTION

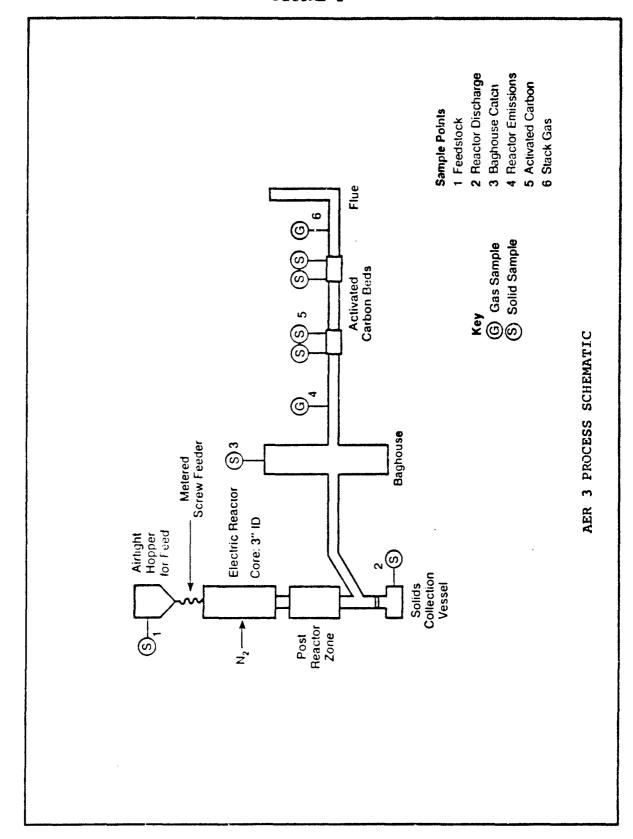
Huber's hazardous waste treatment process is based on the Advanced Electric Reactor (AER) shown in Figure 1.

The reactor employs a new technology to rapidly heat materials to temperatures in the range of 4000°F using intense thermal radiation in the near infrared region. The reactants, which can be gaseous, liquid, or solid form, are isolated from the reactor core walls by means of a gaseous blanket formed by flowing nitrogen radially inward through the porous core walls. Carbon electrodes are heated and in turn heat the reactor core to incandescent so that the heat transfer is accomplished by thermal radiative coupling from the core to the feed materials. The only feed streams to the reactor are the solid, liquid, or gaseous wastes and the blanket gas—nitrogen.

Destruction is accomplished by pyrolysis rather than oxidation; therefore, typical products and by-products produced by incineration such as carbon monoxide, carbon dioxide, and oxides of nitrogen are not formed in significant concentrations. The principal products of soil borne PCB destruction using the Huber process are hydrogen, chlorine, HCl, elemental carbon, and a granular, free-flowing, solid derived material.

Figure 2 is a simplified process diagram of the HTG process as configured for solid hazardous waste destruction. The solid feed stream is introduced at the top of the reactor by means of a





metered screw feeder connecting the airtight feed hopper to the reactor. Nitrogen is introduced primarily at two points in the reactor annulus. Prior to entering the reactor, the nitrogen is preheated up to 1000°F using an electric circulation heater.

The solid feed passes through the reactor where pyrolysis occurs at temperatures between 3500° and 4500°F. After leaving the reactor, the product gas and waste solids pass through a post-reactor treatment zone (PRTZ).

The PRTZ provides for additional residence time but primarily cools the gas to less than 1000°F prior to downstream particulate cleanup.

Solids exiting the PRTZ are collected in a solids collection vessel which is sealed to the atmosphere. Any additional solids in the product gas are removed as the gases enter a bag house. Any residual organics and chlorine are removed by passing the product gas through activated carbon beds just upstream of the emission stack. The extremely small amount of process gas in the Huber system (150 scfm for a 25,000 ton per year plant) makes it economical to use absolute post reactor gas cleaning as is provided by the activated carbon beds. The organic, particulate, and chlorine free product gas composed almost entirely of nitrogen (some moisture) is then emitted to the atmosphere through the process stack.

For the Gulfport test, Huber will use the Toxbuster unit which is the same AER that was used for the on-site demonstration in Times Beach, Missouri. A schematic of the Toxbuster trailer is shown in Figures 3 and 4.

This unit, shown in Photographs 1, 2, 3, and 4, is Huber's smallest reactor and has an inside core diameter of three inches and a heated length of approximately three feet. This unit is installed in a covered truck/trailer to provide mobility and is used for proof-of-concept experiments and on-site demonstrations such as the Gulfport test.

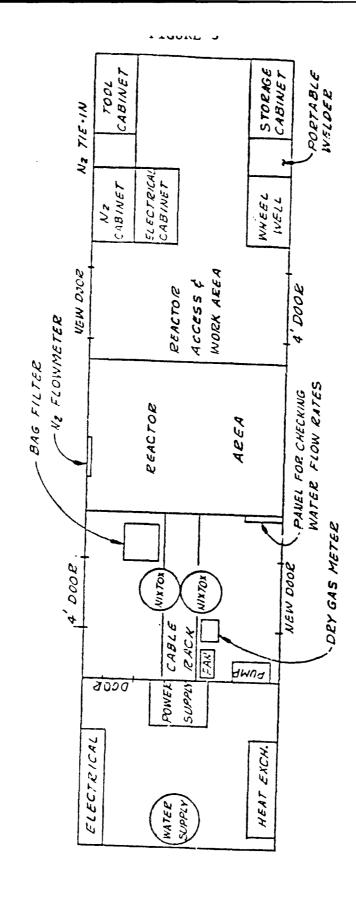
#### SHUTDOWN PROCEDURES

Normal and emergency shutdown of the AER process is relatively simple. Normal and emergency shutdown consists of the following:

- 1. Turn off power to screw feeder.
- Visually verify that no material is being fed to the reactor.
- 3. Close valve which isolates the screw feeder from the reactor.
- Pemove feed tube from reactor and place in a plastic bag (using Level C2 personnel protection).

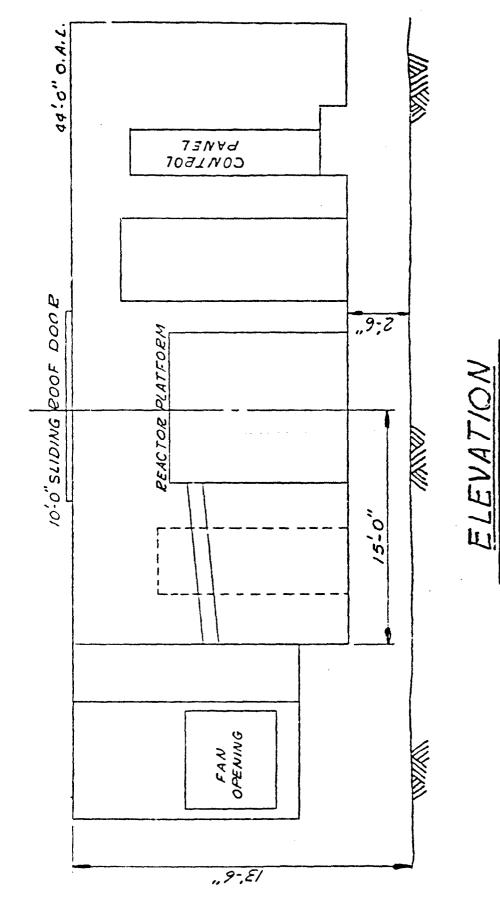
# AEP 3" ELECTRIC REACTOR

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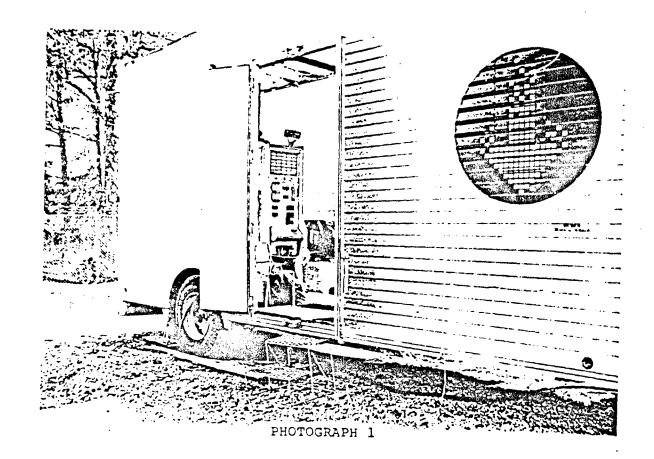
TOP VIEW EQUIPMENT LAYOUT

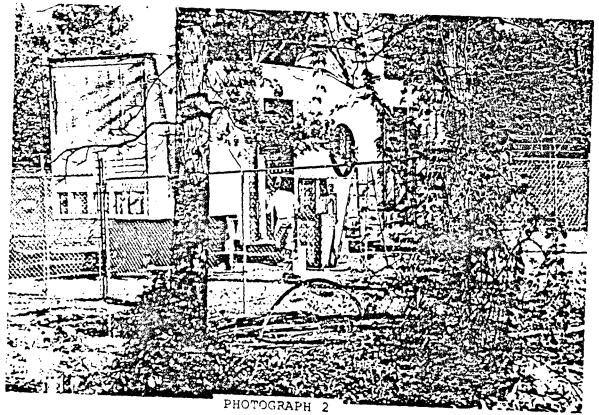
AEP 3" ELECTRIC REACTOR

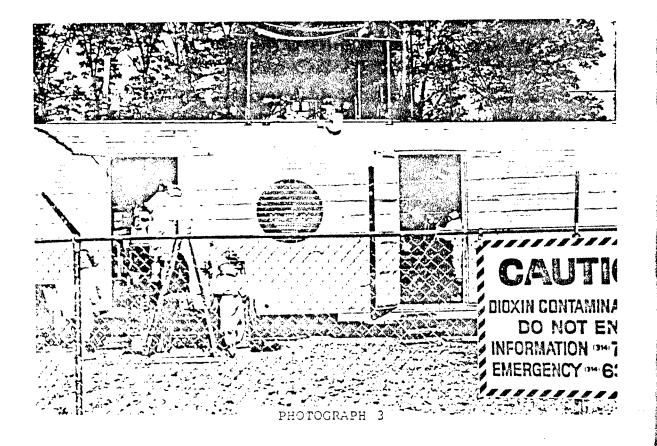


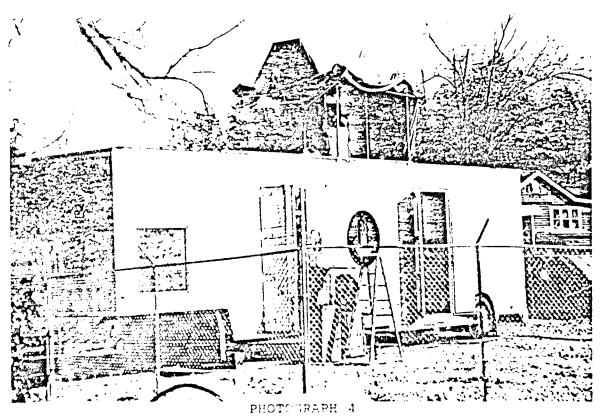
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# 5. Turn off power to reactor.

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The nitrogen purge remains on the reactor until the reactor temperature is about 200°F (normally this will take about three to four hours). After the reactor is cooled to less than 200°F, the nitrogen flow is stopped.

### 3. ACTIVITY SCHEDULE

The current schedule is based upon the Gulfport test being performed in June 1985. Figure 5 highlights the planned activities during June.

During the week of June 10, the Toxbuster will be packed in anticipation of transportation to Gulfport later in that week. Upon arrival at NCBC, the reactor will be located on Lot 42 near the electrical service pole which was installed to provide 480-volt, three-phase service to the Toxbuster. Because purified nitrogen is a necessary part of the AER process, a cryogenic nitrogen trailer will be placed adjacent to the AER. The locations of these units on Lot 42 are shown in Figure 6.

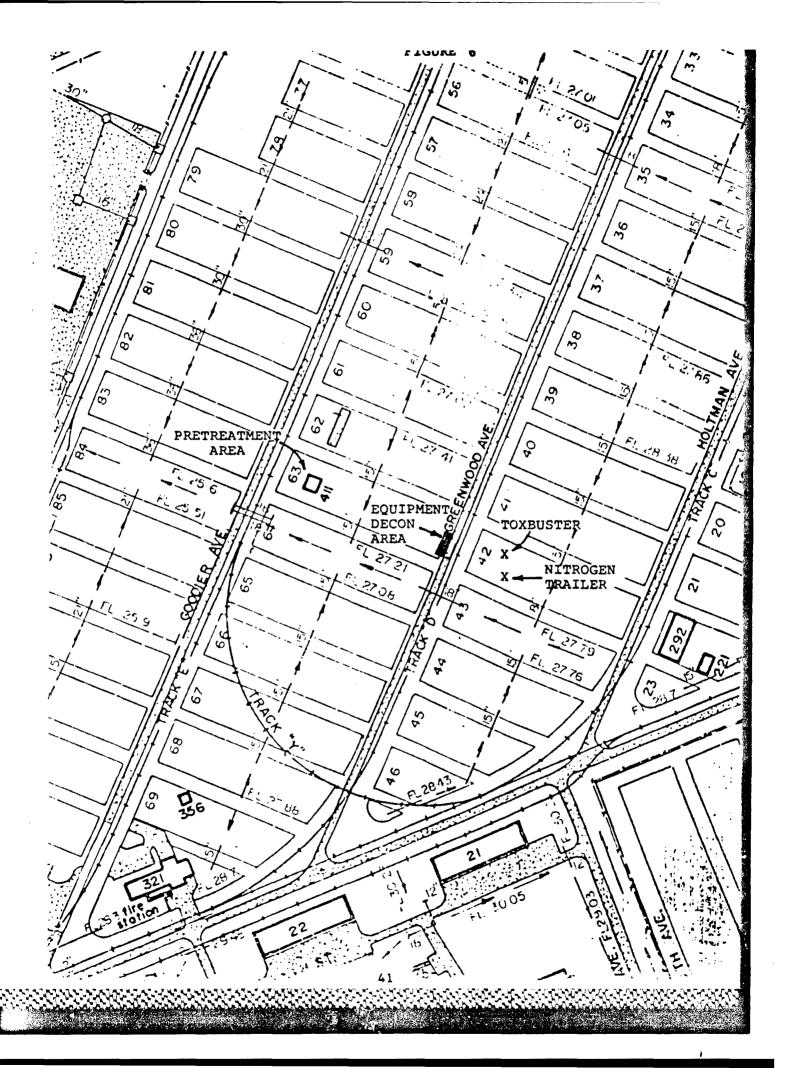
On June 17, the Toxbuster trailer will be leveled, power will be connected, and minor reactor assembly will be performed. Four Huber personnel will be on site to perform these tasks.

On June 18, the feed system will be installed and nitrogen will be hooked up to the trailer in anticipation of a shakedown test on June 19.

During the shakedown test, the Toxbuster will be brought up to approximately 4100°F to identify any problems that may have resulted from transportation of the system. If any repairs are required, they will be performed on June 20 along with other

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PROPOSED GULFPORT DEMONSTRATION TEST, PLAN



remaining test preparations. The remainder of the demonstration personnel will arrive at Gulfport on June 20.

On June 21, final test preparations will be performed. In addition, an overall pretest meeting will be held with all test participants. The agenda for this pretest meeting is shown in Table 1.

The coordination meeting is a necessary function used to ensure that all participants in the test are fully aware of their responsibilities. As shown on the agenda, this will include a brief review of the emergency procedures, health and safety procedures, and run plan. A checklist will also be given to each participant so that he can verify that all of his duties will be performed at the proper time.

Preparation of Gulfport soil to be used in the AER test is being done as a lower tier subcontract by IT Corporation. By June 22, IT Corporation will have the AER feed bin loaded with approximately 1,000 pounds of Gulfport soil that is free-flowing (about 1% moisture content) and ~35 mesh.

On either June 22 or June 23, four individuals will be required to transport the filled feed bin to the AER for installation above the reactor. Prior to transporting the feed bin from the contaminated area, its exterior will be decontaminated. Transportation will require the use of the following personnel:

# TABLE 1

# Agenda For Pretest Meeting

- 1. Introduction and Overview
- 2. Review of Run Plan
- 3. Specific Participant Responsibilities
- 4. Health and Safety Review
- 5. Emergencies and Contingency Plans

- 1. A crane driver.
- An individual to drive a pickup truck in which the feed bin will be transported.
- 3. Two operators in Level C1 protection equipment to install the feed bin on the reactor.

After the feed bin has been set in place above the reactor, the Toxbuster will be off limits (caution tape will be placed on all entrances) to individuals not wearing the following protection equipment (defined as Level C2 protection):

- Standard safety equipment (i.e., hard hat, safety shoes, safety glasses).
- 2. Ventilated Tyvek coveralls.
- 3. Cotton gloves.
- 4. Half-face masks with organic, vapor, and Hepa filter cartridges.

The actual test will begin at 8 a.m. on June 24. Approximately three hours before the test is to begin, the lead operator and process engineer from Team 3 will bring the Toxbuster to operating conditions. The test is scheduled for completion during the afternoon or evening of June 25. Under normal circumstances, the

test should require about 30 to 40 hours to complete; however, unforeseen problems may cause the length of the test to be extended.

Decontamination procedures will begin on June 26. Towards the end of the same day, IT Corporation will take wipe samples for analytical verification that no dioxin is present on any of the AER equipment.

Verification of these tests is expected on Thursday, June 27, for final disassembly and loading of the Toxbuster equipment for its return to Borger on either June 28 or June 29.

### 4. DETAILED RUN PLAN

Attachment 1 is a detailed run plan for the proposed test.

In the event of inclement weather or an emergency, operation of the Toxbuster will cease after emergency shutdown procedures (described in Section 2) are performed. The decision to stop operation of the Toxbuster will be made by the Field Coordinator.

If operation of the Toxbuster is stopped for any reason (i.e., weather problems, equipment breakdown, etc.), the continuation of the test will be determined by the EG&G on-site Project Director.

Attachment 2 summarizes the test conditions. As shown, approximately 1,000 pounds of soil contaminated with Herbicide Orange will be processed. Huber expects between 100-200 parts per billion of 2,3,7,8-tetrachlorodibenzo-dioxin (TCDD) in the feedstock. The concentration of TCDD and other products in the feedstock will be determined by the verification subcontractor.

For the proposed test, the following run conditions will be maintained:

ITEM	VALUE					
Reactor Core Temperature °F	3500-4500					
Nitrogen Rate, scfm	6-10					
Feedstock Charge, Lbs	1,000					
Feed Rate, Lb/Min	0.4-0.6					
Estimated Bag Filter Catch, Lbs	10					

### 5. PROJECT STAFFING

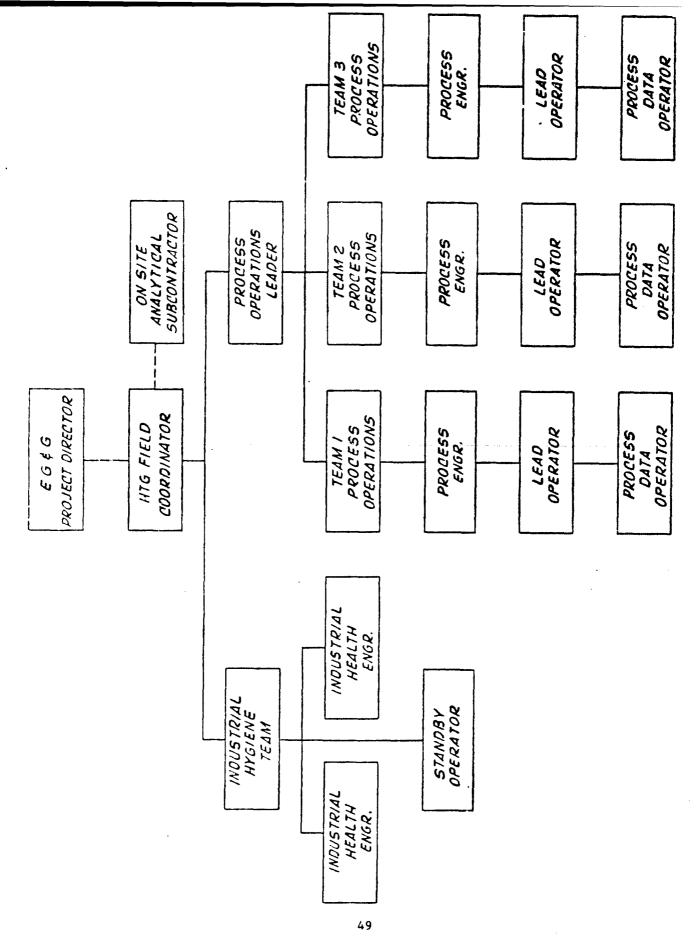
Figure 7 is a field team organizational chart for the personnel involved in the Gulfport test. As shown, this project organization requires six professionals and seven hourly personnel and consists of two groups under the general direction of the field coordinator.

The process operations team, under the direction of the process operations leader, is responsible for the operation of the AER and recording of all process operating parameters.

The industrial hygiene team is responsible for monitoring the activities of the process operations team and ensuring its safe operation.

Because the test is to be performed around the clock, sufficient manpower is required for shift work. The industrial health engineers will work 12-hour shifts during the test.

Attachment 3 is a detailed description of the activities to be performed by all participants during the test.



FIELD TEAM OPGANIZATIONAL MUAD

### 6. FEEDSTOCK DESCRIPTION AND PREPARATION

For proper operation, the AER requires a solid feedstock that has been properly sized and dried. Specifically, this requires solids with a particle distribution near 35 mesh and a moisture content of approximately 1% to ensure a free-flowing product.

IT Corporation has been retained as a lower tier subcontractor to Huber to perform feedstock preparation. Current plans require that IT Corporation complete all feedstock preparations by June 22 and load Huber's feed bin with approximately 1,000 pounds of a free-flowing, -35 mesh mixture of Herbicide Orange contaminated Gulfport soil.

Huber anticipates the following mixture will make up the 1,000 pounds of feedstock provided on June 22.

DESCRIPTION	CONCENTRATION				
Concrete	21%				
Sand/Gravel	45 %				
Shell	31%				
Asphalt	1%				
Tar	2%				

### 7. DATA SAMPLING

During the test, Huber will be gathering information that will describe the operating parameters of the AER process. All sampling and analytical tests will be performed by the verification subcontractor. Huber anticipates a visit (in Borger, Texas) from the verification subcontractor to work out specific equipment needed for performing sampling in Gulfport. Huber will coordinate with the verification subcontractor for obtaining samples during the test. Huber anticipates this will include both solid and gaseous samples.

Attachment 4 is an example of the data sheets to be used by Huber personnel to record process data during the test.

The information obtained from these data sheets will be used along with the analytical data to establish overall destruction efficiencies. The information that will be documented as a result of using these forms includes the following:

- Total Amount of Soil Treated
- Instan neous Soil Feed Rates
- Reactor Core Temperatures (via optical pyrometer)
- PPTZ Temperature (via thermocouple)

- Reactor Volts and Amps
- Nitrogen Flow Rates (using a rotameter)
- Reactor Fower Requirement
- Reactor Pressures
- Total Gas Flow Rate Exiting Process (dry gas meter)

### 8. EQUIPMENT CHECK AND CALIBRATION

Several deliberate measures are used during the operation of the Toxbuster to maintain proper operation. As described in the previous section, several data forms have been used in previous tests for recording process operations parameters. These records were used to record the information in the Times Beach tests and were quite successful. Similar forms will be used during the NCBC tests.

Prior to the NCBC test, the entire AER process flow train will be leak tested using a soap solution. This test is used to verify that all connections are secure and that the entire system is sealed. The following equipment will also be calibrated according to standard calibration procedures:

- Rotameters (using Dry Gas Flow Meter)
- Optical Pyrometer (using portable pyrometer)
- Screw Feeder (by weight loss mechanism and batch testing using a clean surrogate at various settings as a function of time)
- Other Misc. Gauges

Redundancy is built into the AER design and data gathering to ensure proper AER operation. Proper operation of the reactor is

verified by (1) optical pyrometer readings; (2) reactor power consumption; (3) volt and amp readings; and (4) temperature of the treated material. In addition, the nitrogen flow rate is metered at two separate points within the process. The overall nitrogen flow is metered using a dry gas meter as it enters the trailer, and rotameters are used to regulate individual flows.

The flow of soil into the reactor is set by a metered screw feeder which is calibrated prior to all tests. Instantaneous soil feed rates are verified by an in-line weigh scale.

To ensure that all process operating parameters are recorded properly, redundancy is also built into the process data gathering. All process parameter readings are taken by two separate test participants. This procedure is used to guard against deficiencies in the recording of data which serves as a permanent record of the test. In the preparation of the final report, the Project Director (and Operating Team) will review the process operating data and present it in the summary of the test. Additionally, all exceptions to this Test Plan will be identified and discussed.

### 9. DECONTAMINATION

Decontamination procedures following the test are outlined in detail in the Industrial Health Manual and will only be described briefly.

Decontamination of the Toxbuster and associated equipment will be performed on June 26 and 27 according to the present run plan.

Upon completion of the test on June 25, the following feed equipment will be removed by personnel in Level Cl protection (a description of Level Cl personnel protection is included in Huber's Industrial Health Manual) and placed in the decontamination area (located inside the contaminated area):

- Feed Tube
- Feeder
- Feed Bin

This equipment will be decontaminated on either June 25 or 26, depending on how late in the day the test ends. All decontamination will be performed in Level C1 personnel protection. Decontamination will be performed just inside the contaminated zone using either steam cleaning or washing with a trisodium phosphate water solution. After cleaning, the equipment will be placed on plastic (in a buffer zone between the contaminated and

clean zone) and wipe sampled for decontamination verification.

Until the results of the wipe sample are available, the equipment will remain wrapped in plastic in the buffer zone.

After the Toxbuster has been allowed to cool to ambient temperatures, the treated soil collection vessel will be unhooked from the AER process flow train, sampled by the verification subcontractor, sealed, weighed, and placed in the contaminated zone (or buffer zone). Removal of the collection vessel will be performed in Level C1 protection equipment.

Toxbuster trailer, IT Corporation will enter the trailer and obtain wipe samples to verify that no TCDD is present. Wipe samples of the feed equipment will be taken immediately after decontamination procedures.

The results of the Toxbuster interior and feed equipment wipe samples taken on June 25 or 26 will be available on June 26 or 27 so that equipment disassembly and packaging can be performed.

All materials requiring disposal will be placed in fiber drums for disposal by EG&G. In addition, the two 55-gallon drums of activated carbon (Nixtox filters) will be removed from the AER process flow train and given to EG&G for disposal. All residual waters generated during decontamination procedures will be drained on the contaminated test site for evaporation.

# 10. FINAL REPORT

The current schedule requires that Huber prepare a draft final report which will be submitted to EG&G on September 3, 1985. After comments are received from EG&G by September 16, the final report will be issued September 30.

Contained in the final report will be the Collowing items:

- Executive Summary
- Process Description
- Operating Parameters
- Review and Audit of Industrial Hygiene Plan
- On-Site Performance Summary
- Frequency of Sampling and Analytical Procedures Used
- Results and Conclusions

### ATTACHMENT 1

Gulfport Run Plan

# RESEARCH AND TEST EVALUATION GULPPORT RUN PLAN REVISION NO. 2

APRIL 15, 1985

TIME AND DATE

ACTION

6/22/85

Obtain Samples Record weights Preparation

The Process Team will weigh the filter bags and the feed bin and record these weights on the data sheets. The top and bottom of each Nixtox activated carbon drum will be sampled by the verification subcontractor. The feed bin, which will have been filled with about 1,000 pounds of soil by the pretreatment subcontractor (IT Corporation), will be installed over the reactor by test personnel in level "C1" protection. Feedstock samples will be taken by the verification subcontractor prior to feed bin installation. IT Corporation will take two pretest wipe samples. In addition, industrial hygiene monitoring equipment will be set up for sampling during the entire testing period. The equipment decontamination area will be set up for use on 6/26/85.

05:00 6/24/85

Reactor Warmup

The process engineer and lead operator from Team 3 will bring the reactor up to operating conditions by 08:00.

08:00 6/24/85

Begin Test Record process data

The reactor core will be maintained at between 3500 and 4500 F for the test. Ten to twenty minutes is allowed for Process Team 1 to optimize the feed rate and the nitrogen rate. The soil feed rate will begin at about .4 lb/m and be gradually increased tr .6 lb/m by 08:30. This will provide some time for the system to equalize. The control data will be recorded every 15 minutes starting at 08:00 and continuing until shutdown.

08:50

Process gas sampling

The start of emission testing will be determined by the field coordinator. No further process changes will take place (other than emergency shutdown). Occasionally, the soil flow may be stopped (for less than I minute) to periodically clean the feed tube via a closed system in-line brush.

09:25

0<sub>2</sub>, CO<sub>2</sub>, CO, Stack Analyses

The emission testing team will be taking orsat and dfaeger tube samples at the stack. All other functions will continue.

The three process teams will operate the reactor on 8-hour shifts.

14:00 to 24:00 6/25/85

End of Test Samples

The field coordinator will determine the end of the test. The Process Team will remove contaminated equipment and place in decontamination area for cleaning. The feed bin will be weighed before it is decontaminated. The reactor and process components will be allowed to cool overnight. Reactor emissions samples will be taken by the verification subcontractor for later analysis. The industrial hygiene air filters will be turned over to the verification subcontractor for later analysis.

08:00 6/26/85

Samples

The treated solids collection vessel will be removed from the AER, sampled, sealed, weighed, and placed in the contaminated area. The Nixtox drums will be sampled. The bag filter vessel will be dismantled, sampled, and the filter bags will be weighed. All weights will be recorded by the process engineer and lead operator. Bag filter bags and other residue materials will be placed in a fiber drum for disposal.

10:00 6/26/85

Decontamination Samples

The Process Team will decontaminate the feed bin, feed hopper, and feed tube. IT Corporation will take wipe samples on the feed equipment, the "charge end" of the reactor, and the discharge end of the reactor. In addition, four other surfaces will be wipe tested. These wipe samples will be taken by the IT Corporation for immediate analysis to confirm decontamination of the Toxbuster.

13:00

Decontamination Analysis

The Process Team will decontaminate all other Toxbuster equipment under review of the industrial hygiene engineer.

The verification subcontractor will perform the process emissions analyses, activated carbon analyses, and air filters analyses. If any of these are positive for TCDD, the field coordinator will determine if any of the "Priority 3" samples need analysis.

### ATTACHMENT 2

### SUMMARY OF TEST CONDITIONS

- 1. Location: Naval Construction Battalion Center, Gulfport, Mississippi
- Test to be conducted in cooperation with EG&G, Idaho Falls, Idaho.
- 3. Date: Late June, 1985 (nominally June 24, 1985)
- 4. Number of Tests: One (1)
- 5. Feed Rate: Between 0.4 and 0.6 lb/min
- 6. Test Duration: Approximately 30 to 40 hours
- 7. Dioxin Concentration: Approximately 100-200 ppb TCDD
- 8. Total Quantity of Soil to be Tested: Approximately 1,000 pounds
- 9. Total Quantity of Dioxin to be Destroyed: Approximately 2 x  $10^{-4}$  pounds or 0.09 grams TCDD
- 10. Results Expected:
  - a. Greater than 99.9999% DRE or TCDD not detected in gas phase.
  - b. Greater than 99.9999% DRE or TCDD not detected in the solid phase.
- 11. Expected Reporting Date: September 30, 1985

In every previous demonstration test, the safety and hygiene practices which Huber implemented have adequately protected personnel, equipment, and the environment. Therefore, the procedures shown in the safety plan (also enclosed) have been tested and proven effective. Previous tests have verified that during actual operation, Huber's system is completely closed. Therefore, Level Cl protection will be utilized only when the system is open. As presently envisioned, an open system will be said to exist when preparing feed material, filling feed bins, changing the treated soil hopper, and inspecting equipment component internals.

# ATTACHMENT 3

TEST PARTICIPANT DESCRIPTIONS

Job Title: Field Coordinator

Communications Equipment: Portable radio

Duties: The Field Coordinator has the overall responsibility to see that the test is conducted according to the written test plan. This includes coordinating the efforts of the Process Operations Leader and verification subcontractor. The Field Coordinator is also the primary contact between Huber and the Air Force, Navy, and EG&G. The Field Coordinator addresses any unforeseen problems and keeps everyone informed of test status and problems. The Field Coordinator will don Level "C1" personnel protection if needed during decontamination procedures. Otherwise, Level C2 personnel protection will be used.

Job Title: Industrial Hygiene Engineer (IHE)

Communications Equipment: Portable radio

Duties: The IHE has the authority to stop the run if he deems a safety hazard exists. The IHE cooperates with the Field Coordinator and process engineer to see that all personnel are properly clothed with protection equipment and that their jobs are carried out in a safe manner. The IHE periodically walks through the test site and notes any safety-related problems. During the test, Level C2 equipment will be used.

Job Title: Standby Operator (Runner)

Communications Equipment: None

Duties: The primary function of the Standby Operator is to act as a replacement for any of the operators in the event of an illness or injury. When in reserve, the Standby Operator assists the Industrial Hygiene Engineer or will handle miscellaneous duties. During the test, Level C2 equipment will be used.

Job Title: Process Engineers - Process Operations

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Communications Equipment: Radio while at the top of the reactor

Duties: The Process Engineers rotate with the Lead Operator to monitor the process at the top of the reactor through the site glass. During the one-half intervals when not at the top of the reactor, the Process Engineer walks through the test site and monitors the equipment and the operators and lends assistance and direction as necessary. The Process Engineers are responsible for seeing that the operators carry out their job functions as specified in their respec-tive assignment lists. The Process Engineers have complete responsibility for operation of the equipment, documentation of run data, and safety of the operation. The Process Engineers are expected to require Level "C1" uniforms when decontamination procedures are being performed. During the test, Level C2 protective equipment will be worn.

Job Title: Lead Operator

Communications Equipment: Portable radio

The Lead Operator is responsible for the operation of Duties: the reactor and auxiliary equipment under the direction of the Process Engineer. He ensures that all process variables are maintained at prescribed levels and immediately notifies the Process Engineer if there are problems or process conditions vary outside of prescribed limits. The Lead Operator also keeps a log of all process changes and the time each change occurs. By monitoring the controls, he ensures that all equipment is operating properly. In the event of equipment problems, the Lead Operator relays instructions to other operators via radio under the direction of the Process Engineer. The Lead Operators are expected to require Level "C1" uniforms when decontamination procedures are being performed. During the test, Level C2 protective equipment will be worn.

Job Title: Process Data Operator

Communications Equipment: Portable radio

Duties: The Process Data Operator is responsible for real-time monitoring of the process equipment during the run. He takes periodic readings to obtain instantaneous measurements of the feed rate and other process parameters during the test. In addition, he checks for cooling water leaks, nitrogen gas leaks, etc. He immediately reports any conditions that might affect the safe operation of the equipment to the Process Engineer. Much of the data collected by the Process Data Operator is duplication to avoid holes in the overall data for proper documentation. During the test, Level C2 protective equipment will be worn.

### ATTACHMENT 4

Process Data Sheets

# DIOXIN DEMONSTRATION DATA SHEET

# PROCESS LOG

REACTOR: _	DATE:
TEST NO:	TEAM:
TIME	COMMENTS
<del></del>	
	·
	70
SIGNATURE	DATE DATE

Page

DATE: //		
ITEM TIME		
FEED DATA		
Feeder Setting		
Instantaneous Feed Rate(lb/m)		
Total Soil Fed (lbs)		
REACTOR DATA		
Core Temp. Set Point		
II. o		
Millivolt Reading		
Reactor Power, KW		
NITROGEN FLOWS		
Blanket Gas (Upper Plenum) %PSI		
Sweep Gas (Lower Plenum) %PSI		
Radiamatic Purge Gas		
Sight Glass No Purge ( )		
Collection Vessel N. Purge (scfm)		
N2 C		
ון (ד) וו		
•		
מדל המא ווברבד - ניחב המא		
PRT2 DATA		
Upper PRTZ Temp, (T2) °F		
Lower PRTZ Temp. (T3) °F		

DATE:

A B

of

Page

	_					
Upper Plenum •F						
Reactor Voltage:						
ØA - ØB						
GA - ØC						
ØB - ØC						
Reactor Amps:						
ØA				-		
ØB						
ØC						
						,
		ŕ				

Signature(s):

# NCBC GULPPORT DEMONSTRATION DATA SHEET

# - PRETEST/POSTTEST CHECKLIST -

New Filter Bags, Wt: Filter Bags After Test, Wt: Feed Bin Empty, Wt: Feed Bin Full, Wt: Feed Bin After Test, Wt: Collection Vessel Empty, Wt: Collection Vessel After Test, Wt: Fiber Drums Filled After This Test:  PRETEST/POSTTEST SAMPLING CHECKLIST				
Location/Item	<u>Time</u> Sample Obtained	Individual's Initials Taking Custody		
1. Feed Bin 2. Pretest Wipe Samples 3. Nixtox Drums, Before Test 4. Nixtox Drums, After Test 5. Bag Filter Sample 6. Treated Soil Sample 7. Decon Wipe Samples  a. Feed Bin b. Feed Hopper c. Screw Feeder d. Feed Tube e. Inside Trailer				
Comments				
Signature(s)	<u>-</u> Da	te:		

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# APPENDIX E

INDUSTRIAL HYGIENE, HEALTH, AND SAFETY
PLANS SUPPORTING HUBER'S AER DEMONSTRATION
TEST AT NCBC

INDUSTRIAL HYGIENE, HEALTH, AND SAFETY PLAN FOR THE TREATMENT OF SOIL CONTAINING HERBICIDE ORANGE USING THE ADVANCED ELECTRIC REACTOR AT THE NAVAL CONSTRUCTION BATTALION CENTER IN GULFPORT, MISSISSIPPI



# **HUBER TECHNOLOGY**

J. M. HUBER CORPORATION

P. O. Box 2831 Borger, Texas 79008-2831 (806) 274-6331 INDUSTRIAL HYGIENE, HEALTH, AND SAFETY
PLAN FOR THE TREATMENT OF SOIL
CONTAINING HERBICIDE ORANGE USING
THE ADVANCED ELECTRIC REACTOR AT THE
NAVAL CONSTRUCTION BATTALION CENTER
IN GULFPORT, MISSISSIPPI

# Prepared for:

EG&G Idaho, Inc. 1955 Freemont Avenue P.O. Box 1625 Idaho Falls, Idaho 83415

# Prepared by:

Darrell B. Derrington, Jr., P.E., and Jimmy W. Boyd, P.E. Huber Technology Group J. M. Huber Corporation 1100 Penn Avenue Borger, Texas 79007

April 29, 1985

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## Introduction

The J. M. Huber Corporation has successfully and safely tested the use of a new technology for the degradation/destruction of dioxin in soil. This process, an Advanced Electric Reactor, has utilized destroy to laboratory prepared octachlorodibenzo-p-dioxin in soil in two separate tests in Borger, Texas. Additionally, a highly successful and efficient test was recently completed in Times Beach, Missouri detoxifying tetrachlorodibenzo-p-dioxin contaminated soil. This technology has also been demonstrated to be highly effective in the of destruction polychlorinated biphenyls (PCB) hexachlorobenzene (HCB) in soils and other organics such as CCl, as liquids or on soils.

In every previous demonstration test, the safety and hygienic practices which Huber implemented adequately protected personnel, equipment, and the environment. Therefore, the procedures detailed in this safety plan have been tested and proven effective.

The following Industrial Hygiene, Health, and Safety Plan addresses the use of Huber's Advanced Electric Reactor for destruction of dioxin and other similar compounds (products contained in Herbicide Orange) in soil at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi.

## MATERIAL CONTROLS

In order to minimize employees exposure to dioxin, the contaminated soils will be transferred from the ground to the pretreatment facility and then to the reactor in enclosed containers. These will be filled utilizing techniques which prohibit direct personal contact with the material and inhibit fugitive dust emissions.

#### PROCESS CONTROLS

The destruction process is essentially a closed system due to the many safeguards Huber has built into the system. This includes the use of closed feed bins and hoppers for the decontaminated product and carefully fitted connections between the feed bins, reactor product bins, and the air pollution control system (bag filters and activated carbon beds). The closed Huber system also operates at very low pressures (about 1-2 inches of water) and thus offers little potential for hazards in the event of unforeseen operating problems

## TEST AREA CONTROL

The test area will be roped off using caution tape allowing an area sufficient to permit all of the operations which are part of the test to occur unhindered. Access to and departure from the site will be carefully controlled with specifically designated entrances and exits. Entrance into NCBC is controlled by Navy guards, and periodical patrols of the test site will be made by the Navy.

Two levels of projection, Levels C1 and C2, will be utilized at the job site. These levels are defined as follows:

Level Cl

Level C1 protection provides the following:

Personnel are to be qualitatively fitted with a full face air purifying respirator. The fit test procedures includes the use of isoamyl acetate or irritant smoke and will be administered by Huber's Industrial Hygienist prior to the test.

Combination NIOSH/MSHA approved pesticide or organic vapor/highly toxic particulate filter cartridges or canisters will be utilized. See Addendum B for care and cleaning of respirators.

Saran coated Tyvek coveralls with complete coverhood (Ref. 1) will normally be worn. Uncoated Tyvek coveralls may be used if appropriate.\*

VITON gloves - inner

NITRILE gloves or cotton gloves - outer

Rubber boots (decontaminatable) to pull over leather safety shoes or steel-toed rubber boots.

Disposable boots - Latex

Inner cotton garment, either coveralls or long underwear

Sleeves, pant cuffs and coverhoods of coveralls, will be attached to gloves, boots and coveralls, using duct tape.

\* IF AMBIENT WORK STATIONS TEMPERATURE IS ABOVE 70 F AND HEAT BECOMES A PROBLEM, THE SARAN COATED TYVEKS MAY BE REPLACED BY PLAIN TYVEK IN ORDER TO PREVENT ACUTE HEAT STRESS. HUBER'S INDUSTRIAL HYGIENIST ON THE JOB WILL MAKE THIS DETERMINATION.

Level C2 protection includes the use of perforated Tyvek (or similar material) coveralls, rubber boots, cotton gloves, half face masks using organic vapor/highly toxic particulate cartridges (see Addendum B for care and cleaning of respirators), hard hats, and safety glasses.

The equipment will be initially placed on a clean, uncontaminated site situated near the test plot. Since the equipment will also be uncontaminated at this point, all set up and equipment installation will be considered clean and, thus, not require Cl or C2 protection. Standard safety equipment will be used during set up (i.e., hard hat, safety glasses and steel-toed boots).

Previous tests have verified that during actual operation, Huber's system is completely closed. Therefore, Level C1 protection will be utilized only when the system is open. Open systems include preparations of the feed material, filling the feed bin, mounting and unmounting the feed bin on the reactor, changing the solids collection vessel, and inspection of equipment component internals. During normal operation, whenever Level C1 equipment is not required, Level C2 equipment will be used.

Persons and equipment leaving the area will be decontaminated. Extensive equipment decontamination (i.e., steam cleaning) may be foregone if sampling (See Addendum C) shows less than 150 nanograms of dioxin per square meter wipe. The decontamination procedures are listed beginning on page 5.

## Personal Protection

### Training

All project personnel will have been or will be trained regarding the hazards involved in handling Herbicide Orange contaminated soil, use and care of protective equipment, and the Industrial Hygiene and Safety Plan. An additional site specific training course will be provided for employees prior to initiation of site work. Prior to the test, a Safety Meeting will be held to review procedures and key items of the Safety Plan.

## HEAT STRESS

The use of Level C1 protective gear will increase the risk of heat stress. Because of this, a very stringent heat stress monitoring program will be instituted.

Workers in the exclusion zone will work in pairs. "Buddies" must be alert for signs of distress and will frequently check on each other. An additional employee assigned for decontamination will serve as a safety watch. The Safety Watch will be dressed in Level Cl when required. He will monitor the activities of on site persons, watching for signs of heat stress. The Safety Watch will have a buddy who remains in contact with him and is stationed in the non regulated zone.

A shower or hose will be readily available to enable rapid cool down of persons exposed to heat. Huber's Industrial Hygienist will supervise rapid cool-down should it be required.

A break area will be available to allow workers to cope with heat or cold conditions.

Depending on the ambient temperature, a work/rest regimen will be established to enable workers to adjust to heat (See Addendum A). At the discretion of Huber's Industrial Hygienist, work hours may be delayed until ambient temperatures are cooler.

Heat Stress Monitoring specifics are expounded upon in Reference

Prior to workers donning Level C1 personnel protective equipment, the following vital signs will be recorded by Huber's Industrial Hygienist when ambient temperatures are above 70 F.

Weight Heart Rate Oral Temperature Blood Pressure

Heart rate, blood pressure and oral temperature will be monitored each time an individual doffs Level C1 personnel protection when the employee's work site temperature exceeds 70 F. This monitoring will be used along with other criteria to determine rest time and further monitoring needs. The method of decision making for these incidences is shown in Addendum A. Medical data will be collected and interpreted by Huber's Industrial Hygienist.

Addendum A includes a discussion of different heat stresses, monitoring, and protection.

Persons using Level C1 equipment must replace body fluids. As workers doff Level C1 personnel protection they must be encouraged to drink 8 ounces of water, fruit juice or fruit juice flavored water. Commercial electrolyte drinks (for example Gatorade) may be used but the proper electrolyte balance can be achieved by following the above recommendations and by generously adding salt to meals.

## MEDICAL MONITORING

The program in Addendum D will be used for medical monitoring of Huber personnel that use Level Cl protective equipment.

# NOISE

A survey of the test area and trailer carrying the 3" reactor to identify any high noise sources was conducted. Hearing protection is not required unless employees work for more than 1-1/2 hours in the Heat Exchanger Room when the heat exchanger motors are operating.

### DECONTAMINATION

A strictly controlled contamination reduction program has been established for both personnel and equipment. Guidelines are listed below.

Decontamination procedures will be arranged to generate as little excess liquid as possible. Decontamination solution may be a strong trisodium phosphate solution. The solution is prepared by mixing 0.4 pounds of trisodium phosphate into 1 gallon of water. As appropriate, steam cleaning may be used.

# PERSONNEL

Because IT Corporation will already have an employee decontamination facility set up, Huber will utilize IT's facilities for personnel decontamination. A detailed description of IT's employee decontamination facility is contained in IT's Health and Safety Plan.

# EQUIPMENT

Every effort will be made to reduce the need to decontaminate equipment. Small tools, sample containers, etc. will be decontaminated with a damp decon solution wipe off. The procedures used for large equipment decontamination are:

- 1. Purge with clean sand, when possible;
- 2. Scrape clean, and vacuum out all loose material;

- Wash out with trisodium phosphate/water solution using sponge and scrub brush. Sponge rinse with clean water; or
- 4. Steam clean, if required.

Equipment decontamination will be performed on the contaminated site using either a trisodium phosphate solution or a steam generator. After decontamination the cleaned equipment will be placed in a buffer zone (adjacent to the contaminated area) until wipe testing verfies that the equipment is clean. While in the buffer zone, the equipment will be wrapped in plastic.

Huber's recommended acceptable level for contamination reduction on equipment is 150 nanograms of TCDD per square meter wipe sample.

### EMERGENCY RESPONSE PROCEDURES

#### MEDICAL

Any worker suspected of being exposed to dioxin via inhalation, ingestion, or skin contact may be taken immediately, following decontamination, to the designated Medical Facility and will be examined. The need for medical attention will be determined on a case by case basis by Huber's on-site Industrial Hygienist and the J. M. Huber Project Director. Blood and urine samples may be collected and analyzed for appropriate parameters, namely liver function indicators and others as designated by the on-site Industrial Hygienist and the consulting physician.

## SPILL

Any spill or release of dioxin contaminated soil will require remedial action. In the event of a spill or other unanticipated occurrence while test personnel are in Level C2 equipment, the feeder will be turned off and the Toxbuster will be vacated. After donning Level C1 equipment, corrective actions will begin. Small spills will immediately be vacuumed up (using a hazardous waste vacuum cleaner equipped with a HEPA filter) and contaminated surfaces will be washed with soap and water. Large spills, which require more extensive decontamination, will be evaluated by on-site management and appropriate actions followed as the situation directs. Shovels, sand, absorbents, etc., will be maintained on-site to accommodate these situations.

## FIRE

Personnel are trained in use of portable fire extinguishers. These will be maintained on site. The location and use of portable fire extinguishers in the Toxbuster will be reviewed prior to Huber's endurance/preparation run in May. Additional fire reporting, response, and evacuation procedures at the NCBC will be followed. A fire will require use of SCBA for personnel protection.

# SPECIAL SAFETY CONSIDERATIONS

Heat From the Reactors

In addition to ambient heat, there will also be heat generated by the reactors. This will add to the heat burden on the workers, making heat stress monitoring all the more essential when Level Cl equipment is used.

Workers must be carefully observed and monitored.

Some of the reactor associated equipment is also a hot object contact hazard. Because workers may have impaired visibility and

hampered movement due to the personal protection, it is important to remind them of this hazard.

#### FALLS

The reactor must be accessed and attended by ladder and elevated platforms. Safeguards are built into the system following applicable OSHA guidelines, but the use of the personal protection equipment will make these areas and access to them more hazardous. Therefore, workers will be advised of the specific areas where extra precautions are required.

#### ELECTRICAL

High voltage electrical components are safely guarded. However, workers will be reminded of these potential hazards. If there is a need for electrical service during the test, a special protection protocol will be worked out by the Industrial Health Engineer, the project director, and the operators so the electrician will be protected from exposure, but will still be able to work safely with the electrical gear.

# INDUSTRIAL HYGIENE MONITORING

There are no formally established Industrial Hygiene Monitoring procedures yet developed for workers potentially exposed to Herbicide Orange containing dioxins. IT Corporation is responsible for the equipment to be used for the air monitoring program. Huber recommends the use of Industrial Hygiene Monitoring (i.e., personnel and area sampling) to determine airborne indications of potential exposure. Addendum C contains the recommended Industrial Hygiene Monitoring equipment and frequency of sampling. IT Corporation samplers will be placed in the work area by the Huber Project Director or someone he designates and will be placed in locations or on persons most likely to be exposed to dioxin (i.e., near feed bin at top of reactor). Analysis of samples should be a modification of the procedure used for stack sample analysis and wipe sample analysis.

# References

- 1. A proposal to provide support for a Hazardous Waste Treatment Demonstration, prepared for J. M. Huber Corporation, Borger, Texas, Proposal No. P-84-1828, Site Safety Plan, submitted jointly by Roy F. Weston, Inc., West Chester, PA, and York Research Consultants, Denver, CO, 1984.
- 2. The Industrial Environment-its Evaluation and Control, U. S. Department of Health, Education, and Welfare, 1973.
- 3. WESTON-SPER Health and Safety Training Manual Roy F. Weston, Inc., in association with Jacobs Engineering Group Inc., Tetra Tech, Inc., and ICF Incorporated, 1982.

Addendum A

# CLASSIFICATION, MEDICAL ASPECTS, AND PREVENTION OF HEAT ILLNESS

# Introduction

Weather conditions are a serious consideration in planning and conducting site operations. H -, humid weather can cause physical discomfort, loss of efficiency, personal injury, and in the extreme, can be life threatening. The worker is highly susceptible to these effects due to the wearing of protective clothing which decreases natural body ventilation and hence cooling. There are six generally recognized types of heat illness briefly described in decreasing order of severity below.

# Heatstroke and Heat Hyperpyrexia

Clinical Features: Heatstroke: (1) hot dry skin: red, mottled, or cyanotic skin; (2) high rising core temperature, 104.9 F and over; (3) brain mental confusion, disorders: loss consciousness, convulsions, or coma, as core temperature continues to rise. Fatal, if treatment delayed.

> Heat hyperpyrexia: a milder form; core temperature lower; less severe brain disorders; some sweating.

Underlying Physiological Disturbance:

Heatstroke: failure of the central drive for sweating (cause unknown) leading to loss of evaporative cooling and an uncontrolled accelerating rise in core temperature.

hyperpyrexia: partial rather complete failure of sweating.

Predisposing Factors:

(1) sustained exertion in heat by unacclimatized workers, (2) obesity and lack of physical fitness, (3) recent alcohol intake, (4) dehydration, (5) individual susceptibility, (6) chronic cardiovascular disease in the elderly.

Treatment:

Heatstroke: immediate and rapid cooling by immersion in chilled water with massage, or by wrapping in wet sheet with vigorous fanning with cool dry air. Avoid over-cooling. Treat shock if present. Obtain emergency medical care.

Heat hyperpyrexia: less drastic cooling required if sweating still present and core temperature 104.9 F. Obtain emergency medical care.

Prevention:

Medical screening of workers. Selection based on health and physical fitness. Acclimatization for 8 to 14 days by graded work and heat exposure. Monitoring workers during sustained work in severe heat.

# Heat Exhaustion

Clinical Features:

(1) Fatigue, nausea, headache, giddiness; (2) skin clammy and moist, complexion pale, muddy, or with hectic flush; (3) may faint on standing, with rapid thready pulse and low blood pressure; (4) oral temperature normal or low, but rectal temperature usually elevated (37.5 to 38.5 C). Water-restriction type: urine volume small, highly concentrated. Salt-restriction type: urine less concentrated, chlorides less than 3 milligrams per liter.

Underlying
Physiological
Disturbance:

(1) dehydration from deficiency of water and/or salt intake; (2) depletion of circulating blood volume; (3) circulatory strain from competing demands for blood flow to skin and to active muscles.

Predisposing Factors:

(1) sustained exertion in heat; (2) lack of acclimatization; (3) failure to replace water and/or salt lost in sweat.

Treatment:

Remove to cooler environment. Administer salted fluids by mouth or give intravenous infusions of normal saline (0.9 percent) if patient is unconscious or vomiting. Keep at rest until urine volume and salt content indicate that salt and water balances have been restored. Obtain emergency medical care.

Prevention:

Acclimatize workers using a breaking in schedule for 1 or 2 weeks. Supplement dietary salt only during acclimatization. Ample drinking water to be available at all times and to be taken frequently during work day.

Clinical Features: Painful spasms of muscles used during work (arms, legs, or abdominal). Onset during or after work hours.

Underlying Physiological Disturbance: Loss of body salt in sweat. Water intake dilutes electrolytes. Water enters muscles, causing spasms.

Predisposing Factors:

(1) heavy sweating during hot work; (2) drinking large volumes of water without replacing salt loss.

Treatment:

Salted liquids by mouth, or more prompt. Frelief by intravenous infusion, massage the cramped muscles. Obtain medical care as required.

Prevention:

Adequate salt intake with meals. In unacclimatized men, provide salted (0.1 percent) drinking water.

## Heat Syncope

Clinical Features:

Fainting while standing erect and immobile in heat.

Underlying
Physiological
Disturbance:

Pooling of blood in dilated vessels of skin and lower parts of the body.

Predisposing Factors:

Lack of acclimatization.

Treatment:

Remove to cooler area. Recovery prompt and complete.

Prevention:

Acclimatization. Intermittent activity to assist venous return to heart.

# Heat Rash

Clinical Features:

Profuse tiny raised red vesicles (blister like) on affected areas. Pricking sensations during heat exposure.

Underlying Physiological Disturbance: Plugging of sweat gland ducts with retention of sweat and inflammatory reaction.

Predisposing factors:

Unrelieved exposure to humid heat with skin continuously wet with unevaporated sweat.

Treatment:

Mild drying lotions. Skin cleanliness to prevent infection. Obtain medical care as required.

Prevention:

Cooled sleeping quarters to allow skin to dry

between heat exposure.

# Heat Fatigue-Transient

Clinical Features:

Impaired performance of skilled sensorimotor,

mental, or vigilance tasks in heat.

Underlying Physiological Disturbance: Discomfort and physiological strain.

Predisposing:

Performance decrement greater in unaccli-

matized and unskilled men.

Treatment:

Not indicated unless accompanied by other

heat illness.

Prevention:

Acclimatization and training for work in the

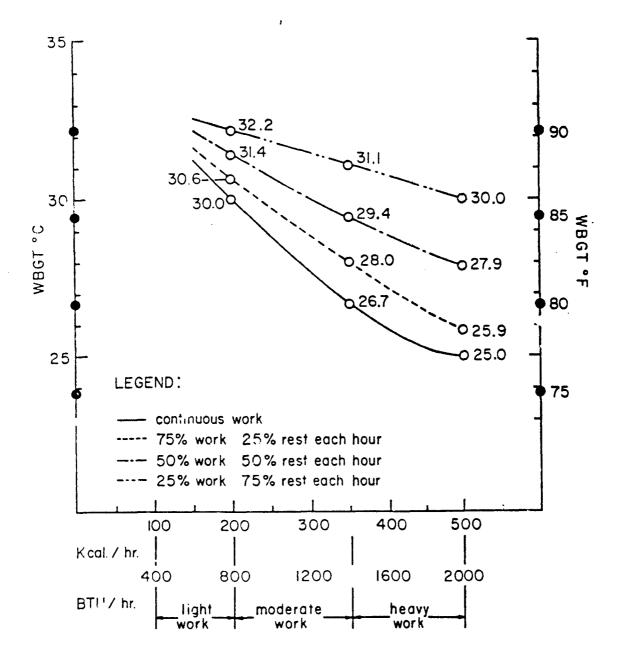
heat.

## Prevention

Heat illness is not 100% preventable; however, by using physically and medically fit workers (no colds, sunburns, etc.), careful planning, work distribution, and personnel monitoring, the likelihood of heat illness can be drastically reduced. A first planning step is to consider the weather conditions and level of protective clothing.

Employees required to use Level C1 protection equipment will be monitored for heat stress by the Wet Bulb Globe Temperature Index (WBGT) technique. This method will require the use of a heat stress monitoring device, such as the Wibget Heat Stress Monitor (Renter Stokes).

The WBGT shall be compared to the Theshold Limit Valves (TLV) outlined in the ACGIH TLV's Manual (see Figure 1 on the following page), and a work-rest regimen established, as necessary, according to the WBGT obtained. Because workers will be wearing impermeable Level C1 protective clothing, 5 C must be subtracted from the TLV's for heat stress.



American Conference of Governmental Industrial Hygienists: Cincinnati, Ohio. 1971.

Figure 1 Permissible Heat Exposure Threshold Limit Value.

Addendum B

### CAFE AND CLEANING OF RESPIPATORS

# I. GENTRAL REQUIPEMENTS

Any organization using respirators on a routive basis should have a process for their date and occasions. The contrast of a process is a contrast that all restinators are majorations at their colonial officiency resear. If they are moduled in any way, their contrastion Captors may be voided. Usually one person in an organization is trained to inspect, plean, repair, and story respirators.

The program should be based on the number and types of respirators, working conditions, and hazards involved. In general, the program should include:

- -instruction
- -Cleaning and Disinfection
- -Repair
- -Storage

## II. INSPECTION

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Inspect respirators after each use. Inspect a respirator that is kept ready for emergency use monthly to assure it will perform satisfactorily.

On air-purifying respirators, thoroughly check all connections for gaskets and "O" rings and for proper tightness. Check the condition of the face piece and all its parts, connecting air tube, and headbands. Inspect rubber or elastomer parts for pliability and signs of deterioration.

Maintain a record for each respirator inspection, including date, inspector, and any unusual conditions or findings.

## III. CLEANING AND DISINFECTION

Collect respirators at a central location. Brief employees required to wear respirators on the respirator program and assure them that they will always receive a clean and sanitized respirator. Such assurances can boost morale. Clean and disinfect respirators as follows:

- -Remove all cartridges, canisters, and filters, plus gaskets or seals not affixed to their seats.
- -Remove elastic headbands.
- -Remove exhalation cover.
- -Remove speaking diaphragm or speaking diaphragm-exhalation valve assembly.

- -Remove inhalation valves.
- -Wash face piece and breathing tube in cleaner/ sanitizer powder mixed with warm water, preferably at 120 to 140 F. Wash components separately from the face mask, as necessary. Remove heavy soil from surfaces with a hand brush.
- -Remove all parts from the wash water and rinse twice in clean warm water.
- -Air dry parts in a designated clean area.
- -Wipe face pieces, valves, and seats with a damp lint-free cloth to remove any remaining soap or other foreign materials.

NOTE: Most respirator manufacturers market their own cleaners/sanitizers as dry mixtures of a bactericidal agent and a mild detergent. One-ounce packets for individual use and bulk packages for quantity use are usually available.

#### IV. REPAIRS

Only a trained person with proper tools and replacement parts should work on respirators. No one should ever attempt to replace components or to make adjustments or repairs beyond the manufacturer's recommendations. It may be necessary to send high-pressure-side components of SCBA's to an authorized facility for repairs. Huber intends to have spare equipment on hand to use in the event of component failure. Repair of respirators will be performed at the factory. In the event that an emergency repair is required, a CIH will make the repairs as follows:

- -Disassemble and hand clean the pressure-demand and exhalation valve assembly (SCBA's only). Exercise care to avoid damage to the rubber diaphragm.
- -Replace all faulty or questionable parts or assemblies. Use parts only specifically designed for the particular respirator.
- -Reassemble the entire respirator and visually inspect the completed assembly.
- -Insert new filters, cartridges, or canisters, as required. Make sure that gaskets or seals are in place and tightly sealed.

#### V. STORAGE

Follow manufacturers' storage instructions, which are always

furnished with new respirators or affixed to the lid of the carrying case. In addition, these general instructions may be helpful:

- -After respirators have been inspected, cleaned, and repaired, store them so to protect against dust, excessive moisture, damaging chemicals, extreme temperatures and direct sunlight.
- -Do not store respirators in clothes lockers, bench drawers, or tool boxes. Place them in wall compartments at work stations or in a work area designated for emergency equipment. Store them in the original carton or carrying case.
- -Draw clean respirators from storage for each use. Each unit can be sealed in a plastic bag, placed in a separate box, and tagged for immediate use.

Addendum C

J. M. HUBER, INC.

Industrial Hygiene Sample Plan

DIOXIN DESTRUCTION TESTS

#### INTRODUCTION

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Huber's required industrial hygiene test plan is designed to give a representative, but thorough testing of both airborne concentrations of dioxin and any surface contamination which may occur during the testing.

## SAMPLE PROCEDURES

Huber recommends that air samples be collected by using personnel or area samplers with a sampling rate of at least 3.0 liters per minute. Any dioxin should be collected on a glass fiber filter. Because TCDD has such a low vapor pressure, very little gaseous phase TCDD is expected. For this reason, sorbent tube samples should be obtained by placing XAD-2 sorbent tubes as a back-up to the glass fiber filter. A sampling time of 7 hours is needed to get the required 1260 liters. The lower detection limit for 1260 liters of air is 0.4 nanograms per cubic meter.

Pretest wipe samples should be collected on the day prior to the Site Test. Wipe area is defined as one square meter of surface area. In instances where the area is less than one square meter, the area of the equipment being tested will be adequate (i.e., wipe down the entire piece of equipment).

The table on the following page indicates the suggested number of samples to be taken and their location. The nature of the samples taken, their location and sampling frequency will be determined by Huber's on-site Industrial Hygienist.

# SUGGESTED INDUSTRIAL HYGIENE SAMPLING PLAN

	PRETEST	TEST	DECON
A. Feed System			3
B. Charge end of reactor		1	
C. Discharge end of reactor 1		1	
D. Air pollution control system- inside first elbow		1	
E. Air pollution control system- <sup>2</sup> discharge		1	
F. Flat surface near reactor charge		1	
G. Flat surface in discharge area 3		1	
H. Additional sample		··1· · ·	
- Pretest wipe sample	1		
- Pretest wipe sample	1		
Air Samples			
A. Hi-vol at control panel		1	
B. Hi-vol at trailer door		1	
C. Personnel at reactor feed		1	
D. Personnel at reactor discharge		1	

# Footnotes:

- Analyze C only if D or F positive.
   Analyze F only if E and G positive.
   Analyze H only if G positive.
   A Process Engineer's respirator filter (picked at random) should be analyzed for the presence of dioxin as he has the greatest potential for exposure.

Addendum D

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SCOOLAN SECRECION DEPOSITIVE ACCESCIONISCOSSISSISSISSISSISSISSES

J. M. HUBER, INC.

Medical Monitoring Program

## Heat Stress Monitoring

For monitoring the body's recuperative ability to excess heat, one or more of the following techniques should be used as a screening mechanism. Monitoring of personnel wearing impervious clothing should commence when the ambient temperature is 70 F or above. Frequency of monitoring should increase as the ambient temperature increases or as slow recovery rates are indicated. When temperatures exceed 85 F, workers should be monitored for heat stress after every work period. The parameters to be monitored, and the frequency of monitoring, will be determined by Huber's Industrial Hygienist.

- 1. Heart rate (HR) should be measured by the radial pulse for 30 seconds as early as possible in the resting period. The HR at the beginning of the rest period should not exceed 110 beats per minute. If the HR is higher, the next work period should be shortened by 10 minutes (or 33%), while the length of the rest period stays the same. If the pulse rate is 100 beats per minute at the beginning of the next rest period, the following work cycle should be shortened by 33%.
- 2. Body temperature should be measured orally with a clinical thermometer as early as possible in the resting period. Oral temperature (OT) at the beginning of the rest period should not exceed 99 F. If it does, the next work period should be shortened by 10 minutes (or 33%), while the length of the rest period stays the same. However, if the OT exceeds 99.7 F at the beginning of the next period, the following work cycle should be further shortened by 33%. OT should be measured again at the end of the rest period to make sure that it has dropped below 99 F.
- Body water loss (BWL) due to sweating should be measured by weighing the worker before and after wearing Level C1 personnel protection. The clothing worn should be similar at both weighings; preferably the worker should be nude. The scale should be accurate to plus or minus 1/4 lb. BWL should not exceed 1.5% of the total body weight. If it does, the worker should be instructed to increase his daily intake of fluids by the weight lost. Ideally, body fluids should be maintained at a constant level during the work day. This requires replacement of salt lost in sweat as well.
- 4. Good hygienic standards must be maintained by frequent change of clothing and daily showering. Clothing should be permitted to dry during rest periods. Persons who notice skin problems should immediately consult medical personnel.

# D. Effects of Heat Stress

If the body's physiological processes fail to maintain a normal body temperature because of excessive heat, a number of physical reactions can occur ranging from mild (such as fatigue, irritability, anxiety, and decreased concentration, dexterity, or movement) to fatal. Standard reference books should be consulted for specific treatment or qualified medical aid sought.

# APPENDIX F

MODIFIED EPA CARCINOGEN ASSESSMENT GROUP'S LIST FOR HERBICIDE ORANGE CONTAMINATION

#### APPENDIX F

# MODIFIED EPA CARCINOGEN ASSESSMENT GROUP'S LIST FOR HERBICIDE ORANGE CONTAMINATION

A copy of the draft "The Carcinogen Assessment Group's List of Carcinogens, July 14, 1980," dated 1984, was obtained from EPA (Headquarters) to review for compounds which could be associated with Merkicide Orange (HO). The Carcinogen Assessment Group's (CAG) list consists of chemicals for which there is substantial evidence of carcinogenicity.

Due to the generality of the CAG list, many of the listed chemicals are not applicable. Based on a screening review by Chemical Sciences personnel at EG&G Idaho, Inc., creosote and identified chemicals that are also on the EPA Priority Pollutant List (PPL) were determined as applicable for analytical testing of the treated NCBC soil to support the soil restoration evaluation. The PPL modified for HO is shown in Appendix G.

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## APPENDIX G

MODIFIED PRIORITY POLLUTANT LIST FOR HERBICIDE ORANGE CONTAMINATION

# APPENDIX G MODIFIED PRIORITY POLLUTANT LIST FOR HERBICIDE ORANGE CONTAMINATION

The Priority Pollutant List (PPL) referenced in Section 307.(a)(1) of the Federal Water Pollution Control Act as amended by the Clean Water Act of 1977 consisted of 129 toxic pollutants or combinations of pollutants. This list was subsequently reduced to 126 by formal deletions presented in the Federal Register (di- and tri-chlorofluoromethanes, January 8, 1981, 46 FR 2266; and bis-(chloromethyl) ether, February 4, 1981, 46 FR 10723).

Based on a review of this list by Chemical Sciences personnel at EG&G Idaho, Inc., the list of 125 priority pollutants in Table G-1 were evaluated as necessary for laboratory analysis of the treated soil to support soil restoration evaluation. The one chemical not included is 1,2-diphenylhydrazine (PPL #37) because its reactivity rules out presence of the chemical because of the time since the HO storage area was used.

The list is organized into six groups; purgeable organics, base/neutral extractable organic compounds, acid extractable organic compounds, pesticides/PCBs, metals, and miscellaneous. The PPL number and chemical analysis serial (CAS) number are included for cross reference.

TABLE G-1. MODIFIED PPL FOR HO CONTAMINATION

Priority Pollutant		CAS
Number	Name	Number
	Purgeable Organics (28)	
	Acrolein	107-02-მ
	Acrylonitrile	107-13-1
4V	Benzene	71-43-2
6V	Carbon tetrachloromethane	56-23-5
	(carbon tetrachloride)	
7V	Chlorobenzene	108-90-7
10 <b>V</b>	1,2-Dichloroethane	107-06-2
J 1 V	1,1,1-Trichloroethane	71-55-6
13V	1,1-Dichloroethane	75-34-3
14V	1,1,2-Trichloroethane	79-00-5
15V	1,1,2,2-Trichloroethane	79-34-5
16V	Chloroethane (ethyl chloride)	75-00-3
19V	2-Chloroethyl vinyl ether	100-75-8
23V	Trichloromethane (chloroform)	67-66-3
29V	1,1-Dichloroethene	75-35-4
30 <b>V</b>	Trans-1,2-dichloroethene	156-60-5
32V	1,2-Dichloropropane	78-87-5
33V	1,3-Dichloropropane	10061-01-05
38V	Ethyl benzene	100-41-4
44V	Dichloromethane (methylene chloride)	75-09-2
44V	Chloromethane (methyl chloride)	
46V	Dromomethane (methyl bromide)	
47V	Tribromomethane (bromoform)	75-25-2
48V	Bromodichloromethane	75-27-4
51 <b>V</b>	Dibromochloromethane	124-48-1
85V	Tetrachloroethene	127-18-4
86V	Tuolene	108-88-3
87V	Trichloroethene	79::01-6
88V	Chloroethene (vinyl chloride)	75-01-4
	Base/Neutral Extractable Organic Compounds (45	5)
1.0	A	
1 B	Acenaphthene	83-32-9
5 B	Benzidine	92-87-5
8B 4R	1,2,4-Trichlorobenzene Newachlorobenzene	120-82-1
9B	Hexachlorobenzene	118-74-1

TABLE G-1. MODIFIED PPL FOR HG CONTAMINATION (CONTINUED)

Priority Pollutant		CAS
Number	Name Name	Number
12B	Hexachloroethane	67-72-1
18B	bis (2-Chloroethyl) ether	111-44-4
20B	2-Chloronaphthalene	91-50-1
25B	1,2-Dichlorubenzene	95-58-1
268	1,3-Dichlorobenzene	541-73-1
27B	1,4-Dichlorobenzene	106-46-7
28B	3,3'-Dichlorobenzidine	91-94-1
33B	2.4-Dinitrotoluene	131-11-3
36B	2,6-Dinitrotoluene	606-20-3
39B	Fluoranthene	206-44-0
40B	4-Chlorophenyl phenyl ether	7005-72 <b>-3</b>
41B	4-Bromophenyl phenyl ether	101-55-3
42B	bis (2-Chloroisopropyl) ether	108-60-1
43B	bis (2-Chloroethoxy) methane	111-91-1
52B	Hexachlorobutadiene	87-68-3
53 <b>B</b>	Hexachlorocyclopentadiene	77-47-4
54B	Isophorone	78-59-1
553	Naphthalene	91-20-3
56B	Nitrobenzene	
62B	Diphenyl nitrosamine	62-75-9
	(N-nitrosodiphenylamine)	
63B	Di-n-propyl nitrosamine	621-64-7
	(N-Nitrosodi-n-propylamine)	
66B	bis (2-Ethylhexyl) phthalate	117-81-7
67B	Benzyl butyl phthalate	85-68-7
68B	Di-n-butyl phthalate	84-74-2
698	Di-n-octyl phathalate	117-84-0
70B	Diethyl phathalate	84-66-2
71B	Dimethyl phathalate	131-11-3
72B	Benzo(a)anthracene	5 <b>6-55-3</b>
73B	Benzo(a)p <b>yrene</b>	50-32-8
74B	Benzo(b)fluoranthene	205-99-2
25B	Benzo(k)fluoranthene	207-08-9
76B	Chrysene	218-01-9

TABLE G-1. MODIFIED PPL FOR HO CONTAMINATION (CONTINUED)

Priority Pollutant		CAS
Number	Name	Number
77B	Acenaphthylene	208-96-8
78B	Anthracene	120-12-7
70B 79B	Benzo(g,n,i)perylene	191-24-2
80B	Fluorene	86-73-7
81B	Phenathrene	81-01-8
828	Dibenzo(a)anthracene	53-70-3
83B	Indeno(1,2,3-c,d)pyrene	193-39-5
84B	Pyrene	129-00-0
	Dimethyl nitrosamine	86-30-6
	(N-nitrosodimethylamine)	
	Acid Extractable Organic Compounds (11)	
		20.24.2
21A	2,4,5-Trichlorophenol	88-06-2
22A	4-Chloro-3-methylphenol	59-50-7
0.4.4	(p-Chlorc-m-cresol)	05.57.0
24A	2-Chlorophenol	95-57-8
31A	2,4-Dichlorophenol	120-83-2
344	2,4-Dimethylphenol	105-67-9
57A	2-Nitrophenol	88-75-2
58A	4-Nitrophenol	100-02-7
59 <b>A</b>	2,4-Dinitrophenol	51-28-5
60 <b>A</b>	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)	534-52-1
64A	Tentachlorophenol	87-86-5
65A	Pheno1	108-95-1
	Pesticides/PCBs (26)	
	Aldrin	309-00-2
	Alpha-BHC	319-84-6
	Beta-BHC	319-85-7
	Gamma-BHC (Lindane)	58-89-9
	Delta-BHC .	319-86-8
	Chlordane	57-74-9
	4,4'-DDD	72-55-8
	4,4 *-DDE	72-54-8
	4,4'-DDT	50-29-3

TABLE G-1. MODIFIED PPL FOR HO CONTAMINATION (CONCLUDED)

Priority Pollutant		CAS
Number	Name	Number
	•	
	Dieldrin	60-57-1
	Endosulfan I	959-98-8
	Endosulfan II	33212-65-9
	Endosulfan suifate	1031-07-8
	Endrin	72-20-8
	Endrin aldehyde	7421-93-4
	Heptachlor	76-44-8
	Heptachlor epoxide	1024-57-3
	Toxaphene	800-35-2
	PCB-1016	12674-11-2
	PCB-1221	1104-28-2
	PCB-1232	11141-16-5
	PCB-1242	53469-21-9
	PCB-1248	12672-29-6
	PCB-1254	11097-69-1
	PCB-1260	11096-82-5

## Metals (13)

Antimony
Arsenic
Beryllium
Cadmium
Chromium

Copper
Lead
Mercury
Nickel
Selenium

Silver
Thallium
Zinc

## Miscellaneous (1)

Total Cyanides

## APPENDIX H

MODIFIED LIST OF COMPOUNDS
INDIGENOUS TO HERBICIDE ORANGE

# APPENDIX H MODIFIED LIST OF COMPOUNDS INDIGENOUS TO HERBICIDE ORANGE

Before the Herbicide Orange (HO) stored at the NCBC was destroyed, samples were taken from drums to determine the chemical composition of HO by each manufacturing source. Analytical results were presented in Aerospace Research Laboratories Report AD-AO11 597, Analytical Methodology for Herbicide Orange. Volume 1: Determination of Chemical Composition, May 1975. While a number of chemicals were common to each manufacturer's HO, some chemicals of low concentration were found unique to a single manufacturer. Based on a review of the report data by Chemical Sciences personnel of EG&G Idaho, Inc., a list of chemicals (Table H-1) was prepared as a spectrum of possible HO chemical constituents in the treated NCBC soil that should be analytically tested for the soil restoration evaluation.

## CHEMICAL COMPOUND

2,4-Dichlorophenol 2,4,6-Trichlorophenol Trichloroanisole Dichloro-Methoxyanisole

Butoxydichlorophenol Butoxytrichlorophenol Butyl-monochlorophenoxyacetate Butyl-dichlorophenoxyacetate

Butyl-trichlorophenoxyacetate Butyl-methoxy-dichlorophenoxyacetate Octyl-dichlorophenoxyacetate Octyl-dichlorophenoxypropinonate

Octyl-trichlorophenoxyacetate 1,1-Dibutoxy-2-trichlorophenoxyethane Octyl-methoxy-dichlorophenoxyacetate Butyl-bis-dichlorophenoxyacetate

Butyl ester of bis trichlorophenoxyacetic acid Butyl ester of trichlorophenoxy-(methoxy-dichlorophenoxy)-acetic acid 2,4-Dichlorophenoxyacetic acid (free acid) 2,4,5-Trichlorophenoxyacetic acid (free acid)

## APPENDIX I

ITC 2,3,7,8-TCDD ANALYTICAL RESULTS FOR AER TREATED SOIL
AND BAGHOUSE FILTER SAMPLES



AUG 1 2 1985

August 6, 1985

Mr. D. Derrington J.M. Huber Corp. P. O. Box 2831 Borger, TX 79008

Dear Mr. Derrington,

Attached, please find the final report for the two soil samples received in our laboratory 6/15/85. These results were reported to the site verbally 6/20/85. Please feel free to contact me regarding questions concerning this data.

Sincerely,

Dennis M. Catalano

Dioxin laboratory Manager

njc

xc. DBD 8-12-85 pl

#### HUBER REPORT

## Summary of Method

Two soil samples were received for the analysis of 2,3,7,8-TCDD. The samples and blank were spiked prior to extraction with an internal standard/surrogate solution containing 50 ng  $^{13}$ C-2,3,7,8-TCDD and 10 ng  $^{37}$ Cl-2,3,7,8-TCDD. The samples were soxhlet extracted, and cleaned up using the EPA reference method described in "The Determination of 2,3,7,8-TCDD From Soil and Sediment," revised September 1983. Extracts were analyzed by GC/MS operating in the selected ion monitoring mode for enhanced sensitivity.

## Sample Preparation

Thirty grams of one soil and 6 grams of the bag fines were weighed into separate jars. The samples were spiked with the internal standard/surrogate mixture and allowed to stand overnight for equilibration. The samples were pretreated with 100 ml 1 N HCl for 1 hour and subsequently filtered through a Buchner Funnel and air dried overnight, followed by a 16 hour soxhlet extraction with Benzene. Thirty grams of NaSO<sub>4</sub> served as the blank. The resulting extracts were filtered into a KD Flask and the volume was reduced to approximately 10 ml and subsequently subjected to liquid column chromatography cleanup.

### Sample Cleanup

To aid in the removal of chemical interferences, the sample and blanks were cleaned up using dual column chromatography consisting of an acid-modified silica gel column followed by a neutral alumina column. Detailed descriptions of these cleanup techniques can be found in Option "A" of the EPA reference stated in the summary section. Final extracts were concentrated to near dryness and raised to 50 µl with 11 ng  $^{13}$ C-2,3,7,8-TCDF which was used as an internal standard.

#### GC/MS Analysis and Results

(1) Isomer specific 2,3,7,8-TCDD

The sample extracts were analyzed using HRGC/LRMS scanning in the selected ion monitoring mode for enhanced sensitivity. The column used for this

isomer specific analysis was a 60 m SP 2330 fused silica column. Before acquisition of the samples, a seven isomer performance mixture containing the six most closely eluting TCDD isomers to 2,3,7,8-TCDD was run. In addition, a five point calibration plot was run in triplicate. The mean response factor obtained from the fifteen point calibration was used for all subsequent calculations. The shift standard, analyzed on the same day as the samples, produced an acceptable response factor within 10% of the fifteen point. Percent recovery is reported by comparing <sup>13</sup>C-TCDD to <sup>13</sup>C-TCDF. Accuracy of the method is obtained by the recovery of <sup>37</sup>C1-TCDD versus <sup>13</sup>C-TCDD.

The results shown in Table 1 are reported in ppb. A detection limit is calculated from 2.5 times the signal in the area of the elution of  $^{13}\text{C-TCDD}$  whenever a sample contains no detectable TCDD.

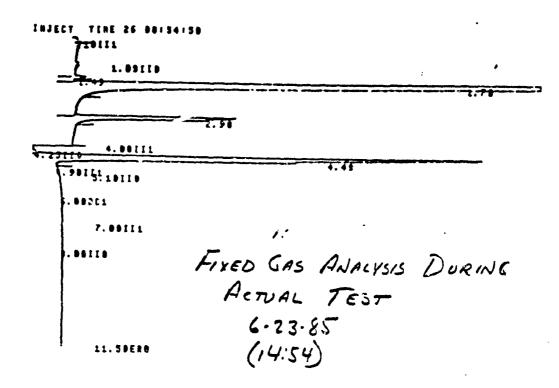
Table 1. Isomer Specific Results

Sample 1D Client #	# 1I	Date	Time	2,3,7,8-TCDD (ppb)	Surrogate (% Accuracy)	% Recovery
Huber Run 1 (bag fines AER)	12609	6/20/85	17:40	0.75	94%	<b>%</b> 06
HU-NCBC-R1-02 (treated soil AER)	J2610	6/20/85	18:18	ND(0.045)	89%	<b>3</b> 866
Reagent Blank	1BLK 381	6/17/85	13:48	ND(0.097)	91%	87%

## APPENDIX J

HUBER FUEL GAS ANALYSIS DATA SHEETS

191799111 6. 88861 . ..... 9. 90II 9 FIRED GAS PUALISIS DURING ACRIPL TEST 6-23-85 (14:20) INDEX AMALYST: R. G. RYAN COHE RT 1.51 3637.579 4.51 352147 UNNORMALIZED TOTAL 96.727 HAME COHC BTU 67 SROSS BTU/CU.FT. DRY 9 14.650 BROSS STU/CU.FT.



日本とこれが経過なるうかなではないのかの場合のないので

いったとうとは関係されていている。

33.55.55

255788555

STATES

FLUE BAS ANALYSIS M. ILE 1 METHOD 5. ANALYST: P. G. RYAN MAME COHE RT RRER BC RF 9.837 94.721 1.171 4.871 1.49 147 #1 4145. 1. 1.141 42 329254 #1 3615.225 Çū 2.98 1596.03 002 4.48 14312 01 3636. TOTALS 100. 347761 UNHORMALIZED TOTAL 96.15 FILE 1 METHOD 5. INDEX HANE CONC Sr. Br. 0.5 HR 0.037 H2 94.721 0. 9161 \* H 4 0. C D 1.171 0.05 4. 871 BROSS BTU/CU.FT. DRY 0 14.696 DRY 0 14.696 68 SPECIFIC GRAVITY 8. 9898355 BROSS BTU/CU.FT. DRY 9 14.65# 3.7472574 SPECIFIC GRAVITY DRY 9 14.658 8. 9867671 BROSS STU/CU.FT. SRT # 14.658

SPECIFIC BRAVETY SAT P 14.630

3.6933347

0. 9725676

```
FIXED GAS ANALYSIS DURAGE
ACTUAL TEST
6.23-85
(16:05)
```

FLUE GAS	ANALYSIS	25 i	1:25:30		
FILE 1	METHOD 5.	RUN 797	IHDEX	3	
AHALYSTI	R. G. RYAH				
MARE	CDHC	RT	ARER BC	RF	RRT
AR H2	0.834 94.788	1.51 1.72	141 01 342549 01	4284. 3637.579	1. 1.139
C D S	1.181 3.993	3.01 4.5	4248 81 14743 81	3614.865 3712.4	1.992 2.98
TOTALS	186.		361673		
UNNORMALI	ZED TOTAL	99.347			
FILE 1	METHOD 5.	<b>9</b> UH 797	IMBEX	3	
HARE	CDMC	stu	Sp. Br.		
H 2 O 2 R 0 H 2 C H 4	B. B. B. B34 94.788	0. 0. 0.	0. 0. 0.8005 0.9168		
50 502 7079L	1.181 3.997 188.	3. B D.	8.8114 8.8687		
GROSS BTE SPECIFIC GROSS BTE SPECIFIC GPOSS BTE SPECIFIC	SRAVITY DRY P J/CU.FT. DPY P SRAVITY DRY P J/CU.FT. SAT P	14.658 14.658 14.658	3.79181 8.9894145 3.7792579 8.9863473 3.7248747 6.9721538		69

## . Ki off Gas tre B. F AER-3 16:09 6-23-85

```
FIXED CAS ANALYSIS DURING
A CTUAL TEST

6-23-85

(16:09)
```

FLUE SAS ANALYSIS 25 11:08:11

FILE 1 METHOD 5. RUN 796 INBEX 2

ANALYST: R. G. RYAN

CONC	<b>RT</b>	AREF	<b>8</b> C	RF	RRT
8.945	1.49	186	11	4284:	1.
94.976	1.71	343977	91	3637.579	1.148
1.166	ī.	4183	11	2614.865	2.813
2.013	4.5	14658	91	3712.4	3.62
180.		361504			•
	8.045 94.976 1.166 2.012	8.845 1.49 94.976 1.71 1.166 3. 2.812 4.5	8.845 1.49 186 94.976 1.71 343877 1.166 3. 4183 2.813 4.5 14858	8.845 1.49 186 91 94.976 1.71 343877 81 1.166 2. 4183 81 2.812 4.5 14858 81	8.845 1.49 186 81 4284; 94.976 1.71 343877 81 3537.579 1.166 2. 4183 81 2614.865 2.812 4.5 14858 81 3712.4

UNHORMALIZED FOTAL 99.303

MARE CONC BTU SP.Br.  #2	FILE L	TETHOD 5.	RUH 796	I H D E X 2	
02	HARE	CONC	BTU	Sp. Br.	
## 8.045	H 2	0.	●.	ð.	
H2 94.976 8. 8.9186 CH4 8. 9. 9. CD 1.166 3.7 9.813 CD2 D.812 8. 9.8579 TOTAL 108.  GROSS BTU/CU.FT. DRY 9 14.696 3.74286 SPECIFIC GRAVITY DRY 9 14.696 8.982436 GROSS BTU/CU.FT. DRY 9 14.696 3.7312571	0.5	●.	₽.	₽.	
CH4 B. B. B. B. CC	A R	8.045	<b>ð</b> .	ð. <b>888</b> 6	
CH4 B. B. B. B. CO CO CONTROL	H 2	94.976	0.	0.5186	
CD 1.166 3.7 0.013 CD2	CH4	<b>.</b>	0.		
CD2		-			
TOTAL 100.  GROSS BTU/CU.FT. DRY P 14.696 3.74286  SPECIFIC GRAVITY DPY P 14.696 0.9884436 GROSS BTU/CU.FT. DRY P 14.650 3.7312571		·			
70 GROSS BTU/CU.FT. DRY P 14.696 3.74286 SPECIFIC GRAVITY DRY P 14.696 0.9884436 GROSS BTU/CU.FT. DRY P 14.650 3.7312571			• •		
GROSS BTU/CU.FT. DRY P 14.696 3.74286 SPECIFIC GRAVITY DPY P 14.696 0.9884436 GROSS BTU/CU.FT. DRY P 14.658 3.7312371	• • • • •	••••		70	٠.
SPECIFIC GRAVITY DPY # 14.696 0.9884436 GROSS BTU/CU.FT. DRY # 14.658 3.7312571			14 686	7 74305	J
6ROSS BTU/CU.FT. DRY # 14.658 3.7312571					
	SPECIFIC	SERVITY DRY P	14,696	8.9884436	
ABBARA ABBURAN ABBURAN ABBURAN ABBARAN	GROSS BTI	J/CU.FT. DRY #	14.658	3.7312571	
SPECIFIC EPAVITY DRY D 14.650 9.9853794	SPECIFIC	SPAVITY DRY P	14.658	9. 9853794	
68088 BTU/CU.FT. SAT 0 14.650 2.6775647					
SPECIFIC BRAVITY SAT 0 14.658 0.9711999	*LEPIPIT	BANALIA 241 A	14.550	0.7/11777	

## APPENDIX K

IT CORPORATION INDUSTRIAL HYGIENE REPORT FOR ACTIVITIES.
SUPPORTING AER3 NCBC TESTS



AUG 26 1985

August 20, 1935

Mr. Darrell Derrington J. M. Huber Corporation P.O. Box 2831 Borger, Texas 7900-2831

Dear Darrell:

Please find attached a table which gives results of industrial hygiene monitoring for 2,3,7,8 TCDD on June 22, 1985 during the test run of the AER at Gulfport, Mississippi. As can be seen, both employee exposure results were below detectable levels, which shows that the protection outlined in the Health and Safety Plan and worn during on-site activities was appropriate. Also, the area sample was below detectable levels which indicates that there were no fugitive emissions of 2,3,7,8 TCDD from the system.

The following is a general outline of the health and safety items on this job:

- Heat stress readings using the WBGT methods, supplemental with blood pressure, pulse, and oral temperature readings, were taken throughout the test run. The WBGT readings ranged from 84°F-90°F. Appropriate breaks were scheduled and employees took these breaks. No evidence of a heat stress related illness, by observations or physical monitoring, was noted.
- 2. During the actual run the health and safety plan was followed without exception. CI protection was worn when any internal activity was done on the AER, employee decontamination procedures were followed, and other general safety guidelines were practiced.

Regional Office
IT Corporation • 312 Directors Drive • Knoxville, Tennessee 37923 • 615-690-3211

Page 2 Mr. Darrell Derrington August 20, 1985

3. The decontamination plan for equipment was not followed. Due to this, there was potential for contamination of other equipment that had been decontaminated, contamination of clean areas, and undue potential exposure for employees. Several adjustments have to be made in order to assure that the equipment was properly decontaminated.

If you have any questions, please call me.

Sincerly,

Harry A. Pullum, CIH

Manager, Health and Safety

jn

# INDUSTRIAL HYGIENE SAMPLES Huber AER Test Run

g	n - Doseton
None detected at 750 pg None detected	6/22/85

tab: IIAS-knoxville Case No.: Air Force Hatch Snipment No.: Golfport IH

ر د چ			ng/83/rp	ng/sarple TCDU	Instr.			Rel. Ion Abund.	Abund.	S Add	PPB Surroyate		Rela	Relative ion Approance	JURGANCE		,	
	Cleanup	Wet Ht. (9)	:kas.	7	2	Date	Time	320/325	332/334	Meas.	1 Acc'y	320	322	257	328ª	332	334	Contract
34013	Yes	Ξ	9	1.3	4500	6/04/85	12:48		0 x 3	191	, a				247036	9000		ľ
24013	. Yes	Ξ	C.	0.35	4500	6/03/85	12:54		0.87	a.v	q V				34/0/0	80866	01/2/11	ъ.
41017	les	Ξ	2	0.67	4500	6/14/85	13:25		83.0	1 70	£ £				909170	9171/	2/9/9	: 0
24015	Yes	Ξ	E	1.7	4500	6/03/85	13:33			1 1 2	G 6				201160	80508	11/5930	o,
91016	Yes	Ξ	5	0.76	4500	6/04/85	14:02				. d				202102	2/9969	658304	۰ م
71040	res	=	<b>70</b>		45.00	6/04/85	14:47	0.78	8.5	0	0 42	216669	26.4024	100320	333040	602808	068501	Φ,
11018	res	Ξ	55		4500	6/03/85	15:54	0.67	0.82	172	: S	451072	70407	02620	#0/262	80789	266701	: ص
61010	Yes	Ξ	1.0		45.00	6/03/85	15:16	0.85	20.0	7/2	e f	1000	13004	2/2022	20200	607.704	0.000	× 0x
14120	Yes	Ξ	02 02	0.61	45.43	6/03/85	14:37		. 6	: :	â	07601	12304	7110	191/00	40//46	016651	-
17010	ĭe,	Ξ	QN	0.95	<b>2</b> 5(6)	6/04/35	11.18		5	2.2	5 5				3/1312	984840	0/55021	==
34022	Yes	Ξ	3	1.2	45.00	6/21/85	12:29		9.00	0/1	6 6				285346	737096	696680	72
14073	Yes	Ξ	3	0.82	4500	6/21/85	11:05		0.82	25	2 5				26//35	81077	64946	_
34,024	Yes	Ξ	1.2		45.30	6/21/85	17:11	0.85	98	1 10	ζ.	40401	23001	0000	001010	\$1659A	11/9/90	20
J4025	Yes		5.9		45(8)	6/01/85	13.47	98	50.0	7 7		00001	90201	4032	0818/7	701341	858445	_
34656	Yes	Ξ	5	0.58	4500	6/21/85	14:37		9.0	601	60	02664	00066	15757	165692	698760	849682	ထ
34027	Yes	Ξ	S.	0.12	4500	6/21/85	10:29		9	, o	20.2				327880	,63404	964752	20
	105	Ξ	70		45.00	6/24/85	14:22	0.83	3	1	≨ ∓	405050	20000	0.0000	357.55	92248	1102/2	<u>~</u>
520 <b>5</b>	Yes	Ξ	CN	0.63	4503	6/21/85	23:01		8.0	7	1.0	00606	066600	068242	Icaco2	967999	820320	эō:
	res	Ξ	ž	0.60	4500	6/27/85	18:56		8 6		: 3				100/12	0//545	7/05/0	÷ .
34031	Yes	Ξ	5	.0.75	4500	6/27/85	19:46		70.0	2 2	2 3				301376	164377	941600	_
24032	Yes	Ξ.	S.	1.3	4500	6/30/85	21:21		7.0	1 1 1	00				74444	86606	1113610	2
	res	Ξ	Š	1.7	4500	6/04/85	10.43			7.7	20				1/0104	418503	524/28	9
1d:ank 352		Ξ	2	9.	4500	6/03/85	10.01		50.0	2/1	00				296144	789136	968688	Š
	res	Ξ	710	-	4500	6/23/85	20:05		0.00	27	85				395526	1058350	1293060	\$ ∙
									;	•	:				751067	2/3/34	113094	æ

Corrected for contribution by native ICUD; 0.9% of m/z 322 subtracted.

hecovery of spike (13c-1000) on 1H tube - 511.

<sup>C</sup>Recovery of spike ( $^{13}$ C-ICUU) on IH tube \* 6/1.

decovery of spike ( $^{13}$ -ICUU) on IH tube = 683.

MB - Reagent blank
P - Partial Scan/Confirmatory Analysis ND - Fot Detected
N - Nutive ICUD Spike
D - Duplicate/Fortified Field blank

**D357:UC-AF-12** 

## APPENDIX L

MEDICAL EXAMINATION CERTIFICATION FOR AER OPERATING PERSONNEL

## J. M. Huber Corporation

P. O. Box 2831

Borger, Texas 79007

#06) 274-633| Telet 73:8458

September 6, 1985

Harry D. Williams EG&G Idaho, Inc. P. O. Box 1625 Idaho Falls, Idaho 83415

Dear Harry:

Enclosed is a letter from Dr. Richard Rehm certifying that there were no significant medical changes in the Huber personnel who participated in the Gulfport demonstration at the Naval Command Battalion Center Site. As the letter indicates, he found no evidence of any toxic material exposure or ill effects from this operation.

I trust that this will be sufficient information for vour records. Should you have any questions, please feel free to contact me.

Sincerely,

Jimmy WV Boyd, P.E.

Manager Environmental Compliance

pjh

Enclosure

xc: Jack Clem

EG&G File

## AMARILLO INDUSTRIAL HEALTH CENTER 2400 LINE AVENUE AMARILLO, TEXAS 79106

MSSOCIATES
PETER G. FAGAN, M.D.
RICHARD D. REHM, M.D.

TELEPHONE AC 806-372-1135

September 3, 1985

Mr. Dick Shaw J. M. Huber Corp. P.O. Box 2831 Borger, TX 79007

Dear Mr. Shaw:

I have completed reviewing the laboratory reports, histories, and photographs of those Huber employees who were involved in the Gulf port operation. Comparing these results to those of the initial examinations, I do not find any significant medical changes. There is no evidence of any toxic material exposure or any ill effects from this operation.

Once again, we enjoy the opportunity to work with Huber and its employees. Thank you.

Sincerely,

Richard D. Rehm, M.D.

RR/kd

CC: Darrell Derrington

## APPENDIX M

ITC WIPE SAMPLING PROCEDURES

#### APPENDIX M

#### ITC WIPE SAMPLING PROCEDURES

The standard operating procedures established for collecting wet wipe samples are as follows:

- a. Materials and Apparatus
  - 3 inch x 3 inch sterile cotton gauze pads, individually wrapped
  - Sample bottles, glass with Teflon-lined caps
  - Hexane (pesticide grade)
  - Distilled water
  - Glass graduated cylinder
  - Sample labels
  - Sample log and chain-of-custody records
  - Indelible ink pen
  - Ruler or square area guide
  - Tape or other marking material to outline wipe area
- b. Select area for collecting a series of wet wipe samples for a matrix type. Ensure surface area is sufficient to collect all required samples.
- c. Mark the location of the wipe on the item(s).
- d. To collect a wipe sample, use the following procedure:
  - Put on a clean pair of disposable gloves
  - Remove a pad from its individually wrapped package
  - Hold pad in hand
  - Soak pad with 8 ml of hexane using the graduated cylinder to measure volume. Fill cylinder from laboratory squeeze bottle.

- e. Sample an area by applying pressure to the pad, then drawing it across the area in both directions, ensuring that the entire area is well contacted.
- f. Upon completion of the wet wipe sample, carefully fold the pad over at least twice, being careful not to touch the contaminated side of the wipe pad, and place in labeled sample collection bottle. Bottles should be temporarily stored in plastic bags until all samples have been collected.
- g. The sampling person in charge of field data should ensure the following information is accurately recorded:
  - Sample description/item description
  - Sample date and time
  - Area date and time
  - Area sampled
  - Observations/problems, if pertinent
  - Names of sampling personnel
- h. Change gloves after taking each sample.
- i. Upon removal of samples from the site, a Chain-of-Custody form shall be established for the samples. The Chain-of-Custody will act as a transmittal form from sampling personnel to laboratory personnel and will be signed at this time to document that samples are properly delivered and received by appropriate staff members.

## APPENDIX N

# COPIES OF UNIFORM HAZARDOUS WASTE SHIPMENT FORMS FOR SHIPMENTS THAT INCLUDED DIOXIN-CONTAMINATED WASTES FROM AER3 NCBC TESTS

			rage
Exhibit	1.	Waste Agreement Letter	151
Exhibit	2.	Uniform Hazardous Waste Manifest, June 6, 1985	154
Exhibit	3.	Uniform Hazardous Waste Manifest, June 28, 1985	156



## ecology and environment, inc.

195 SUGG ROAD, P.O. BOX D, BUFFALO, NEW YORK 14225, TEL. 716-632-4491, TELEX 91-9183

International Specialists in the Environment

May 22, 1985

Naval Construction Battalion Center Code Orange Gulfport, Mississippi 39501 ATTN: Harry Williams 864-0056

Dear Mr. Williams:

Ecology and Environment, Inc. (E & E) has been conducting sampling at the Naval Construction Battalion Center and Eglin Air Force Base for dioxin under a contract to EG&G Idaho, Inc. During this sampling, sampling wastes consisting of protective clothing, sampling equipment (spoons, drills, aluminum trays, etc.), and drill borings (soil) have been generated. The sampling wastes were packaged in fiber drum containers and left on the respective sites. These wastes are now being processed for disposal by Rollins Environmental Services, Inc.

Paragraph 6 of Rollins form 101-81 (attached) presents a list of chemicals unacceptable to Rollins. None of the chemicals listed in paragraph 6 are in the fiber drums filled by E & E. It is further noted that the requirement for steel drums has been changed by Rollins to fiber drums.

Sincerely,

Louis W. Adams Project Manager

Lin Cicion

## in

## Rollins Environmental Services my Inc.

P.O. Box 808, Deer Pork, Texas 77536 (713) 679-6001

101-81

Con ordinal adult to

RES	Ref.	No.	
	****		

This letter, 29	con receipt	by Rollins Env	vironmental Services (T	X) Inc. ("RES"), o	f your accepta	nce, shall be th	he agreement bet	ween
(defined below),	term, price	and represents	ations					
MARRAHI	ILS. Toc	omply with all	l existing laws, ordinan	ces and regulations	of the United S	tates and of ar	y state, county, 1	lown-
	1 1		at the second section of the second	بدينا بتستسيطه المشاهبين			i necate	

WARRAM - ICES. To comply with all existing laws, ordinances and regulations of the United States and of any state, county, township or municipal subdivision thereof, or other governmental agency which may be applicable to the removal of Waste, RES shall obtain all permits, increases and other forms of documentation required in order to comply with such laws and regulations.

RES INDEMNIFICATION. Following loading and departure from Company's plant, if RES provides transportation or, following delivery file by RES' facility, if Company provides transportation, Company shall be relieved of responsibility and RES shall become solely responsible for any and all loss, damage or injury to persons or property and RES shall indemnify and hold Company harmless from any and all bability, damages, costs, claims, demands, and expenses of whatever type or nature, including, but not limited to, pollution or when damage, which shall be caused by, arise out of, or in any manner be connected with the Waste, except as provided in COMPANY INDEMNIFICATION below.

COMPALL WARRANTS. Company represents and warrants that the Waste loaded and removed under this Agreement shall be the Waste defined on Schedule "A", attached hereto and made a part hereof, and has been thoroughly characterized on the waste data sheet submitted to RES. Company agrees to prepare and execute RES' waste data sheet for each shipment of Waste. If the Waste is packaged, Company warrants that such Waste shall be prepared for shipment and packaged in containers specified by the then current and applicable complations of the United States Department of Transportation, Environmental Protection Agency or any successors the containers and/or any state, municipal and/or Federal agency having jurisdiction, as the case may be. Company shall be responsible for probleged Waste on RES' trailers if RES is providing transportation.

damage or undue wear and tear to equipment, claims, suits, or costs which shall arise or grow out of any injury to any person or persons or any property (including the person or property of Company or its employees) caused by or resulting in any way from Company's failure to comply with Company's Warranty concerning the Waste. Company shall be responsible for and indemnify RES ugainst any and off hability, damages, costs, claims, demands, and expenses of whatever type or nature resulting from the acts and/or omissions of Company and/or its employees, until departure of RES vehicles from Company's plant, if RES provides transportation or if Company prevaides transportation, until delivery f.o.b. RES' facility.

- I do like the right of either party to terminate this Agreement at any time upon thirty (30) days prior written notice. This Agreement shall automatically terminate on
- 2. PANMS STERES shall invoice Company for the hauling and treatment of Waste at the rates and terms set forth on Schedule "A" it is not have an identified part hereof. RES shall add an amount equal to one and one-half percent (12%) or the maximum legally peranasistic to a subsequent shorty (30) day period that such invoice remains unpaid.
- 3 RES SCHEMION. Company understands and agrees that RES, upon notice to Company, has the absolute and unqualified right to reject one on, ment of Waste which does not conform to the description of Schedule "A" (the "Waste Data Sheet") supplied by Company in disk. After any such rejection, RES will, with Company's assistance and approval, pursue all other reasonable means of disposal. If the Waste is rejected, Company shall be obligated (a) to pay the cost of transportation to RES' facility if such transportation was reduced as a relative to Company's premises (Company having the restance of the carrier) and (c) to pay all other reasonable charges incurred by RES with the prior consent of Company.
- RES' facility, if Company provides transportation, Company's plant, if RES provides transportation or, following delivery f.o.b. RES' facility, if Company provides transportation, Company shall be relieved of title responsibility and risk of loss for the Waste, and RES shall take title, responsibility and risk of loss. However, title, risk of loss and all other incidents of ownership to non-conforming Waste shall be accorded to revest in the Company at the time revocation of acceptance is communicated to Company and RES shall be reasonable for its own negligence or willful acts.

FORCE MADEURE. Delays or failure of either party in the performance of its required obligations shall be excused if caused by electronstances beyond the reasonable control of the party affected, including but not limited to, acts of God, strikes, labor holiday, fire, fleod, wandstorm, explosion, riot, war, sabotage, action or request of governmental authority, accident, inability to obtain material, equipment or transportation, provided that a prompt notice of such delay is given and the parties shall be diligent in attempting to remove such cause(s)

152

those specified below:	
Piacetylaminofluorene, Chemical Abstracts Service Registry No. 62759	1
alpha-naphthylamine, Chemical Abstracts Service Registry No. 134327	Y.
odiphenyl, Chemical Abstracts Service Registry No. 9267)	01%
ine, Chemical Abstracts Registry No. 92875	0 15
held-naphthylamine, Chemical Abstracts Service Registry No. 91598	010
heta-propiolactone, Chemical Abstracts Service Registry No. 57578	1:2
bis-chloromethyl ether, Chemical Abstracts Service Registry No. 542881	0.15
3,3'-dichlorobenzidine, Chemical Abstracts Service Registry No. 91941, and its salts	177
4-dimethylaminoazobenzene, Chemical Abstracts Service Registry No. 60117	15.
ethyleneimine, Chemical Abstracts Service Registry No. 151564	10
methyl chloromethyl ether, Chemical Abstracts Service Registry No. 107302	0.17
4,4'-rnethylene bis (2-chloroaniline), Chemical Abstracts Service Registry No. 101144	1/2
4-nitrobiphenyl, Chemical Abstracts Service Registry No. 92933	0.15
N-nitrosodimethylamine, Chemical Abstracts Service Registry No. 62759	177
polychlorinated biphenyls	0.0057
	0.003,1

Additions may be made by RES to the foregoing list of substances from time to time, such additions by RES becoming effective and binding after three days' written notice to Comapny.

Company agrees that all Waste containing asbestos (including actinolite, amosite, anthophyllite, chrysotile, crocidolite, and tremolite) fibers longer than 5 micrometers detectable by phase contrast microscopy shall be subject to the following conditions:

a. The presence of asbestos in the Waste shall be clearly noted on RES' waste data sheet.

b. Waste shall be packaged in closed steel drums bearing a label which conforms with 29 CFR 1910.1001.

Company further represents and warrants that, to the best of its knowledge, Waste does not contain vinyl chloride monomer in a liquid or gaseous form except as specified on RES' waste data sheet.

All previous representations, including but not limited to, proposal(s), purchase order(s) and/or invoice(s), either written or oral are hereby annulled and superseded. No modification shall be binding unless in writing and executed by RES and Company.

Please indicate your agreement to the above recitals by executing and returning a copy of this letter.

ACCEPTED this 10 day of June 19 85	ROLLINS ENVIRONMENTAL SERVICES (TX) INC. ("RE						
HOAFESC /ROVW ("Company")	BY:						
Address ext USAF RSC	Address: P.O. Box 609 Deer Park, Texas 77536						
Protest Manager							

Copy available to DTIC does not permit fully legible reproduction

Exhibit 2

	print or type (Form designed for use on elite (12-pitch) typewriter.)					Form Approved. OMB No. 2000-0404. Expires 7-31-80									
TĽ		NIFORM HAZARDOUS 1. Generator's US EPA ID No. MS 2170022626			Manifest Document No. EG1050-3							in the shaded areas od by Federal Isw.			
3.	Unite	Generator's Name and Mailing Address United States Navy					A. State Manifest Document Number								
4.	Naval Construction Battalion Center Gulfport, MS 39501 Generalors Phone (601) 865-2484						B. State Generator's ID 99928								
5.	Transporte	r 1 Company Name State Motor Tr			EPA ID Numbe 95038998	k*	C. State Transporters ID  D. Transporter's Phone 800-641-758								
7.					EPA ID Numbe						041	-/30			
9					US EPA ID Number			F. Transporter's Phone G. State Facility's ID							
	9. Designated Facility Name and Site Address Rollins Environmental Services 2027 Battleground Road				US EFA ID NUMBER			H. Facility's Phone				The said the said to			
	Deer Park, Texas 71536 TXD 055141378						(713) 479-6001								
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	2027 Bettleground Road Seer Park, Texas 71536	1 TXD 055141378		H. F	acility's Phone (723) 479	. <i>്</i> ീരി		
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## APPENDIX O

ECOLOGY AND ENVIRONMENT, INC. SAMPLING PROTOCOL FOR AER3 NCBC TEST

#### APPENDIX O

#### SAMPLING PROTOCOL

A bound sampling logbook will be individually assigned to each site. The logbook will be kept by the on-site sampling team. Field data sheets will also be used. At a minimum, the following information will be kept in the logbook and on the field data sheets:

- o Site name
- o Demonstration project name
- o Test run number
- o Sample test point number
- o Sample number
- o Date and time sampled
- Ambient air temperature
- o General weather conditions
- o Name of sampler
- o Name of laboratory performing the analysis
- o Date sample was shipped
- o Airbill number.

To enhance decontamination, a small plastic bag will be placed around the outside of the simple bottles and held in place by a rubber band so that any sample spilled during the collection or compositing process will not contaminate the outside of the jar. After sample collection, the sample bottles will be decontaminated by first removing and discarding the rubber band and outer plastic bag. The sample bottle will be washed with clean water, dried, and placed in a clean bag; then the bag will be sealed.

Soil samples will be collected in disposable aluminum trays, using standess steel scoops or spoons. Enough soil will be collected to fill two 16-ounce wide-mouth jars. Pretreated soils will be sampled as they are ted into the furnace feed bins. Treated soil exits the furnace at extremely high temperatures and will be sampled after it has cooled to

ambient air temperature. All soil samples will be composites of at least five aliquots. They will be sieved into the wide-mouth jars through 10-mm screen and the jars will be sealed with aluminum lined caps.

Representative carbon filter bed samples will be collected during the equipment purging and cleaning cycle. They will be placed in two 16-ounce wide-mouth jars and sealed with aluminum-lined caps.

## APPENDIX P

## PACKING LISTS FOR HUBER TEST-RELATED SAMPLES BEING SHIPPED TO ANALYTICAL LABORATORIES

					Page
Exhibit	1.	Cooler	No.	5	165
Exhibit	2.	Cooler	No.	7	168
Exhibit	3.	Cooler	No.	11	172
Exhibit	4.	Cooler	No.	12	176

## PACKING LIST

Cooler No.: NCBC #5
Date Shipped: 6/17/85

Federal Express Airbill No.: 353-895-964

TO:

California Analytical Laboratories, Inc.

2544 Industrial Blvd. West Sacreamento, CA 95691

FROM:

USAF Sampling & Analytical Program Environmental Restoration & Technology Research & Test Evaluation Project

EG&G Idaho, Inc. Code Orange/USAF Project Trailer Naval Construction Battalion Center Gulfport, MS 39501 601/864-0056

Ref:

RFP # C85-130686

Quant.

Sample # / Description

```
IT NCBC-R1-04 / IT PHOTOLYSIS RUN 1 SOLVENT AFTER TREATMENT
IT-NCBC-k2-03 / IT PHOTOLYSIS RUN 2 SOLVENT BEFORE TREATMENT
IT-NCBC-R2-09A / IT PHOTOLYSIS RUN 2 REAR PART OF 1st GAS CARBON
IT-NCBC-R2-27 / IT STACK TEST IN-LINE PARTICULATE FILTER
IT-NCBC-R2-28 / IT STACK TEST FIELD BLANK PARTICULATE FILTER
IT-NCBC-R2-35 / IT STACK TEST KOH (IMPINGER #3)
IT-NCBC-R2-36 / IT STACK TEST KOH FIELD BLANK
EE-NCBC-R1-01 / HI-VOL SAMPLER #1, RUN 1 OFF-SITE CONTROL FILTER
EE-NCBC-R1-02 / HI-VOL SAMPLER #2, RUN 1 ON-SITE CONTROL FILTER
EE-NCBC-R1-03 / HI-VOL SAMPLER #3, RUN 1 ON-SITE DOWNWIND FILTER
EE-NCBC-R1-04 / HI-VOL SAMPLER #4, RUN 1 OFF-SITE DOWNWIND FILTER
```

11 TOTAL SAMPLES

Sample # IT-NCBC-R1-04 taken 6/7/85 at 0430. 1, 1/2 gallon jug. No preservatives.

Analyze for: 2,3,7,8-TCDD DL  $\langle = 0.1 ppb.$ 

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  $DL \leftarrow 0.1 \text{ ppb}$ .

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organic indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Suspended solids content.

Sample # IT-NCBC-R2-03 taken 6/14/85 at 1800. 1, 16 oz jar. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Sample # IT-NCBC-R2-09A taken 6/13/85 at 1950. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  $DL \leftarrow 0.1 \text{ ppb}$ .

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above test result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Total amount of carbon.

Sample #'s IT-NCBC-R2-27 and IT-NCBC-R2-28 taken between 2300 on 6/12/85 and 0500 on 6/13/85, 1, 16 oz jar each. 1, petri dish each. No preservatives. IT-NCBC-R2-27 has an initial tare of 0.2533 g. IT-NCBC-R2-28 has an initial tare of 0.2543 g.

Analyze for: Particulate loading DL <= 0.0001 gr/scf.

Sample #'s IT-NCBC-R2-35 and IT-NCBC-R2-36 taken between 2300 on 6/12/85 and 0500 on 6/13/85. 1, 16 oz jar each. No preservatives.

Analyze for: Hydrogen chloride DL < = 1.0 ppm.

Sample #'s EE-NCBC-R1-01, EE-NCBC-R1-02, EE-NCBC-R1-03, and EE-NCBC-R1-04. Each contains a plastic bag with a particulate filter in a folder. Initial tares are listed below:

```
EE-NCBC-RI-01 (filter # 1131): 3.03360 g.
EE-NCBC-RI-02 (filter # 1132): 3.02484 g.
EE-NCBC-RI-03 (filter # 1133): 2.98913 g.
EE-NCBC-RI-04 (filter # 1134): 3.03537 g.
```

Volumes of air passing through each filter are as follows:

```
EE-NCBC-R1-01: 4845.99 cu m

EE-NCBC-R1-02: 4635.54 cu m (estimate)

EE-NCBC-R1-03: 4845.99 cu m

EE-NCBC-R1-04: 2877.06 cu m
```

Analyze for: Total suspended particulates.

2,3,7,8-TCDD DL < = 0.1 ppb.

£3

### PACKING LIST

Cooler No.: NCBC #7
Date Shipped: 6/17/85

Federal Express Airbill No.: 353-895-964

TO:

California Analytical Laboratories, Inc.

2544 Industrial Blvd. West Sacramento, CA 95691

FROM:

USAF Sampling & Analytical Program Environmental Restoration & Technology Research & Test Evaluation Project

EG&G Idaho, Inc. Code Orange/USAF Project Trailer Naval Construction Battalion Center Gulfport, MS 39501 601/C64-0056

Ref:

RFF # C85-130686

Quant. Sample # / Description

HU-NCBC-R1-02 / HUBER RUN 1 SOIL AFTER TREATMENT
IT-NCBC-R2-02 / IT RUN 2 SOIL AFTER TREATMENT
IT-NCBC-R2-09 / IT RUN 2 FRONT PART OF 1st GAS CARBON BED
HU-NCBC-R1-09 / HUBER RUN 1 FIRST GAS CARBON DRUM

HU-NCBC-RI-09A / HUBER RUN I SECOND (DOWNSTREAM) GAS CARBON DRUM

IT-NCBC-R3-02 / IT RUN 3 SOIL AFTER TREATMENT

TOTAL SAMPLES

Sample HU-NCBC-R1-02 taken 7/14/85 at 0220. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-[CDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # IT-NCBC-R2-02 taken 6/13/85 at 0624. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < -0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # IT-NCBC-R2-09 taken 6/13/85 at 1950. Contains 2, 16 oz jars. No preservatív

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Sample # HU-NCBC-R1-09 taken 6/14/85 at 0315. Contains 2, 16 oz bottles. No preserva

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-,  $\hat{\epsilon}$  hexa-chloro-dibenzofurans DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Hydrogen chloride DL < = 1 ppm.

Nitrogen oxide DL < = 1 ppm.

Sample # HU-NCBC-R1-09A taken 6/14/85 at 0315. Contains 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = ! ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Hydrogen chloride DL < = 1 ppm.

Nitrogen exide DL < = 1 ppm.

Sample # IT-NCBC-R3-02 taken 6/14/85 at 0330. Contains 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL  $\langle = 0.1 \text{ ppb.} \rangle$ 

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins DL  $\prime$  = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

## Packing List

Cooler No. 11

Date Shipped: 6/28/85

Federal Express Airbill No.: 353 895 942

TO:

California Analytical Laboratories, Inc.

2544 Industrial Blvd. West Sacramento, CA 95691

FROM:

14

USAF Sampling & Analytic Program

Environmental Restoration & Technology Research & Test Evaluation Project

EG&G Idaho, Inc.
Code Orange/USAF Project Trailer
Naval Construction Battalion Center
Gulfport, MS 39501
601/864-0056

Ref: RFP # C85-130-130686

Quant. Sample # / Description

TOTAL SAMPLES

```
HU-NCBC-R1-01 / SOIL BEFORE DESTRUCTION PROC. (HUBER RUN #1)
HU-NCBC-R2-01 / SOIL BEFORE DESTRUCTION PROC. (HUBER RUN #2)
HU-NCBC-R2-02 / SOIL AFTER DESTRUCTION PROC. (HUBER RUN #2)
HU-NCBC-R2-09 / HUBER RUN #2 FIRST GAS DRUM
HU-NCBC-R2-09 / HUBER RUN #2 SECOND GAS DRUM
HU-NCBC-R2-09 / HUBER RUN #2 BAGHOUSE PARTICULATE
EE-NCBC-R2-01 / HI VOL. SAMPLER #1, RUN #2 OFF-SITE CONTROL
EE-NCBC-R2-02 / HI VOL. SAMPLER #2, RUN #2 ON-SITE CONTROL
EE-NCBC-R2-03 / HI VOL. SAMPLER #3, RUN #2 ON-SITE DOWNWIND
EE-NCBC-R2-04 / HI VOL. SAMPLER #4, RUN #2 OFF-SITE DOWNWIND
EE-NCBC-R3-01 / HI VOL. SAMPLER #1, RUN #3 OFF-SITE CONTROL
EE-NCBC-R3-02 / HI VOL. SAMPLER #2, RUN #3 ON-SITE CONTROL
EE-NCBC-R3-03 / HI VOL. SAMPLER #3, RUN #3 ON-SITE DOWNWIND
EE-NCBC-R3-04 / HI VOL. SAMPLER #4, RUN #3 OFF-SITE DOWNWIND
```

Sample HU-NCBC-R1-01 taken 6/12/85 at 2135. 2, 16 oz jars. No preservatives.

Sample HU-NCBC-R2-01 taken 6/21/85 at 0130. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chloro-dibenzo p-dioxins, to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to  $DL < or = 0.1 \ ppb$ .

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL< or = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) to LD, or = 10 ppb.

Sample HU-NCBC-R2-92 taken 6/24/85 at 1910. 2, 16 oz jars. No preservatives.

Sample HU-NC3C-R2-03 taken 6/24/85 at 2020. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chlora-dibenzo p-dioxins to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to  $DL < or = 0.1 \ ppb.$ 

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppm.

Organics indigenous to herbacide orange (Appendix D) to a DL < or = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # No's EE-NCBC-R2-01, EE-NCBC-R2-02, EE-NCBC-R2-03, EE-NCBC-R2-04, EE-NCBC-R3-01, EE-NCBC-R3-02, EE-NCBC-R3-03, EE-NCBC-R3-04, each consist of a particulate filter in a folder in a plastic bag. Initial tares are listed below.

Sample No.	Filter No.	Initial Tare	Volume Sampled
EE-NCBC-R2-01	1135	3.01042	4,547.10 <sup>a</sup>
EE-NCBC-R2-02	1136	3.01013	4,637.07 (est.)
E-NCBC-R2-03	1137	2.92856	4,547.10 <sup>a</sup>
EE-NCBC-R2-04	1138	2.96794	4,721.23 <sup>b</sup>
EE-NCBC-R3-01	1139	2.96849	1,761.05
EE-NCBC-R3-02	1140	2.95780	1,596.16 (est.)
EE-NCBC-R3-03	1141	2.97475	1,761.05
EE-NCBC-R3-04	1143	1.97590	1,828.50

- a. Later found in error; corrected value is 4562.
- b. Later found in error; corrected value is 4735.

### Analyze Above Samples For:

Total suspended particulates 2,3,7,8-TCDD to DL < or =0.1 ppb.

Sample HU-NCBC-R2-09 taken 6/24/85 at 1940. 2, 16 oz jars. No preservatives.

Sample HU-NCBC-R2-09A taken 6/24/85 at 1900. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chlorodibenzo-p-dioxins. To DL< or = 0.1 ppb..

Total isomers of tetra-, penta-, and hexa-dibenzofurans to  $DL < or = 0.1 \ ppb$ .

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppb.

lotal amount of carbon present.

Hydrogen chloride to DL < or = 1 ppm.

Nitrogen oxidu to DL < or = 1 ppm.

## PACKING LIST

Cooler No. 12

Date Shipped: 6/28/85

Federal Express Airbill No. 353-895-920

ro:

Battelle Columbus Laboratories, Inc.

Attn: Dr. David Miller

505 King Ave.

Columbus, OH 43201

FROM:

USAF Sampling & Analytic Program

Environmental Restoration & Technology

Research & Test Evaluation Project

EG&G Idaho, Inc. Code Orange / USAF Project Trailer Naval Construction Battalion Center Gulfport, MS 39501 601/864-0056

Quant.	Sample # / Description	
1	HU-NCBC-R2-02/HUBER RUN #2, SOIL AFTER TREATMENT	
1	HU-NCBC-R1-01/HUBER RUN #1, SOIL BEFORE TREATMENT	
1	IT-NCBC-R2-04/IT RUN #2 SOLVENT AFTER PHOTOLYSIS	
3	TOTAL SAMPLES	

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chloro-dibenzo-p-dioxins to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to  $DL \le or = 0.1 \text{ ppb.}$ 

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < cr = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) to DL < or = 10 ppb.

#### In addition:

Sample HU-NCBC-R2-02 taken 6/24/85 at 1910. 2, 16 oz jars. No preservatives. Requires that if any of the above tests results in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), an EP Toxicity Test should be run.

Sample IT-NCRC-R2-04 taken 6/15/85 at 0315. 1, 80 oz amber jug. Non preserved. Requires suspended solids analyses.

Sample HU-NCBC-R1-01 take: 6/12/85 at 2135. 2, 16 oz jars. Non-preserved. Requires no additional analyses.

## APPENDIX Q

REVIEW/EVALUATION OF ANALYTICAL RESULTS FOR HUBER PROCESS VERIFICATION SAMPLES

#### APPENDIX Q

# REVIEW/EVALUATION OF THE ANALYTICAL RESULTS FOR HUBER PROCESS VERIFICATION SAMPLES

Chemical Sciences of EG&G Idaho, Inc., had the responsibility for reviewing and evaluating all analytical data from the J. M. Huber Company process technology demonstration at the NCBC site. California Analytical Laboratories, Inc., (CAL) was selected as the subcontract laboratory for analysis of all process verification samples collected during the Huber demonstration. These samples were shipped to CAL from the NCBC site in several separate batches. Samples were received at the laboratory on June 15, June 21, and June 29, 1985.

The analytical results were transmitted by CAL in several submittals. The various submittals were as follows:

<u>ltem</u>	Date of Submittal	Description
1	9/10/85	Preliminary reports on polychlorinated dibenzo-p-dioxins and dibenzofurans
2	1/21/86	Data summaries and information on analytical protocols
3	1/31/86	Results of sample submitted for EP Toxicity Test
4	3/11/86	Additional data and information plus clarification of data summaries. (This information was provided in response to requests made by EG&G Idaho during meetings with CAL on March 4 and 5, 1986.)
5	3/21/86	Additional information including a revised inorganics analysis data package, some reanalysis results for specific samples, and additional supporting information. (This information was also provided in response to EG&G Idaho requests made during the March 4 and 5, 1986, meetings with CAL.)
6	4/22/86	Results of the reanalysis of five samples for semivolatile organics.

All data submitted by CAL were included in the EG&G Idaho review/evaluation process.

After the samples were submitted to CAL, 2 to 4 months elapsed before any analyses were performed. Thus, sample holding times as dictated by EPA were exceeded by wide margins. Other problems with various portions of the CAL data are documented and discussed in detail in this appendix.

Furthermore, at the time of review, there were no universally accepted data review protocols for the polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). Therefore, because of the problems and limitations of the CAL data and also the absence, in some cases, of applicable data review protocols to validate the results, EG&G Idaho considered it inappropriate to attempt to validate the results in the strict sense used by the EPA. Instead, Chemical Sciences reviewed and evaluated the results to determine if the appropriate analytical protocols were used and applied correctly, if the various calculations were correct, and if the results were consistent and logical.

Results of the EG&G Idaho review/evaluation process are presented in the following sections. Discussion has been broken down by the class of analysis performed, i.e., inorganics, volatile organics.

#### INORGANIC RESULTS

Samples from the Huber demonstration project were submitted to Galifornia Analytical Laboratory for inorganic element analysis, including cymorde, on June 21 and 29, 1985. The methods used for analysis were taken from the U.S. EPA Contract Laboratory Program (CLP) protocols. The specific instrumental techniques used are specified on the data reporting sheets.

On January 21, 1986, analytical results for inorganic elements were submitted to EG&G Idaho for review. A review of the data showed them to be in error. It appears that on the summary sheets dated January 21, 1986, the simplicate results, percent recovery, spiked sample results and relative

percent difference (RPD) values obtained on October 18, 1985, were used. In each case, however, sample results obtained on a different run were used. Therefore, none of the calculations were correct.

The above errors were pointed out to the contract laboratory, and corrected data sheets were prepared on March 19, 1986. All calculations on the new data sheets were correct. From the data submitted, it appears that proper analytical procedures were used.

As stated previously, the samples for inorganic analysis were submitted to the contract laboratory on June 21 and 29, 1985. These samples were analyzed on October 18, 1985. During this time, approximately 5 months, the samples were stored at ambient temperature and without preservatives.

The CLP protocol states that samples for cyanide analysis are to be stored at 4°C and the maximum holding time is 14 days. For mercury, the maximum holding time is 30 days. For all other metals, the maximum holding time is 6 months. It also states that for mercury and all other metals, the pH of the sample is to be adjusted to 2, with nitric acid for preservation purposes. In the protocol, no differentiation is made between liquid and solid samples.

Although the values for metals and cyanide cannot be validated, they can be used as a general guide for evaluating their fate in the Huber process.

Another area of concern is spike recovery values. Each sample was spiked with a known amount of each element being determined. The percent recovery of the spiked elements gives a measure of the extraction efficiency. The percent recovery of each spiked element should fall within 75 to 125 percent of the amount added to the sample. A review of the data shows that 22 spike recovery results, representing 36.5 percent of the values reported, were outside this target window. Twenty-one of the values were low, ranging from 0 to 74 percent spike recovery. The 0 percent

recovery result was for antimony on sample HU-NCBC-R2-09. The high result was for lead on sample HU-NCBC-R2-01. This spike recovery value was reported as 148 percent. CAL's position is that these questionable spike recovery values are normal and are completely acceptable to EPA.

EG&G Idaho contacted the organization performing review of inorganic analytical results for EPA and was informed that currently no action is taken if the spike recovery values are outside the stated limits. However, EG&G Idaho feels that it is indicative of questionable analytical techniques.

#### VOLATILE ORGANIC COMPOUNDS

Various soil and carbon filter samples were analyzed for volatile organic compounds. The analytical procedures were taken from the CLP protocol an' based on EPA Method 624.

The samples submitted to CAL were received by that laboratory on June 21 and 29, 1985. The samples were stored at ambient temperature and extracted on September 16, 1985, approximately 3 months after receipt. In addition, the samples for volatile organic analysis were taken about 3 weeks after the containers had been opened to take samples for semivolatile organic components.

CLP protocol states that samples for volatile organic analysis must be protected from the light and refrigerated at 4°C from the time of receipt until they are extracted. The extraction and analysis are to be done within 10 days of sample receipt.

CLP protocol further states that a 4- or 5-point calibration curve is to be prepared for each instrument used in the analysis. This calibration curve is necessary to determine the linearity of response for that instrument. No evidence could be found that any calibration curve had been prepared.

Based on all of the above, results obtained for volatile organic components are not considered valid.

#### SEMIVOLATILE ORGANIC COMPOUNDS

The semivolatile organic compounds were analyzed using the CLP protocol, which is based on EPA Method 625. The samples submitted to CAL were received at that laborator; on June 21 and 29, 1985. The extraction for semivolatile organic components was performed on August 27, 1985, approximately 2 months after receipt of the samples.

The CLP protocol states that samples for organic analysis must be protected from light and refrigerated at 4°C from the time of receipt until extracted. Solid samples must be extracted within 10 days of receipt and the extract analyzed within 40 days of extraction. The samples in question were stored at ambient temperature for 2 months prior to extraction.

As discussed with the volatile organics, preparation of a 4- or 5-point calibration curve for the analytical instrumentation used is not evident.

The analysis contract required that semivolatile organic compounds be analyzed to 1 ppm. It appears that the contract laboratory arbitrarily decided whether a given sample was analyzed as low concentration or medium concentration. On this basis, the following samples (HU-R1-02, HU-R2-02, and HU-R2-03) required reanalysis. The reanalysis was performed by taking the sample extract from a previous analysis and concentrating the extract from approximately 0.5 g/1 mL to 0.5 g/0.5 mL. Although this procedure should double the concentration and lower the detection limit by half, the reported detection limits were lowered by a factor of four. There was no evidence that sample injection size had been increased. To attain the resorted detection limits, the sample injection size would have to double. One other point of concern is that the detection limit is based on the peak height versus background noise level for any given component. It is

therefore highly improbable that each component would have the same detection limit. However, the factor of four improvement in detection level was reported for all compounds.

Based on all of the above, the reported data cannot be validated. However, because of the lower volatility of semivolatile organic compounds, the length of storage time and storage conditions would not be as critical as for the volatile organic compounds. Therefore, the values obtained can probably be used as a guide for evaluating the Huber process.

#### PESTICIDE/PCB ANALYSIS

The pesticide analysis includes the chlorinated insecticides and herbicides as well as the polychlorinated biphenyls. These components were contermined using the CLP protocol, which is based on EPA Method 608. One method covers both pesticides and PCBs.

The same storage conditions and time restrictions for sample extraction and analysis exist for the pesticide/PCB samples as for the semivolatile organic material. Like the other organics, a calibration curve is not evident.

Based on the above, the reported pesticide/PCB values cannot be validated. However, like the semivolatile organic components and for the same reason, the reported data can possibly be used as a guide to evaluate the Huber process.

#### DIOXIN AND DIBENZOFURAN RESULTS

Two types of analyses were performed for the specified PCDDs and PCDFs: total isomer class content for tetra, penta, and hexachlorinated PCDDs and PCDFs and 2,3,7,8-TCDD isomer-specific. The review methodology was to evaluate all data in terms of applicable ion ratios, retention times, and signal-to-noise ratios to determine if the analytical results were correctly interpreted.

The isomer-specific 2,5,7,8-TCDD data were examined and evaluated, using the same criteria applied during the soil sampling and analysis program conducted previously for the USAF at the NCBC site. These criteria are detailed in the EPA document for reviewing 2,3,7,8-TCDD analytical results (Reference 1), and the criteria are listed in the annex to this appendix. The results of the evaluation will be discussed in two parts, the isomer class analyses and the isomer specific analysis. The isomer class analyses will be discussed first.

In its original proposal to EG&G Idaho, California Analytical Laboratories proposed to use EPA Method 8280 (as modified by EMSL-Las Vegas) to perform the isomer class analyses. CAL stipulated that extraction methodologies would probably require modification because of the types of sample matrices involved, and this was understood by EG&G Idaho. However, it was not felt that the modification in extraction methodology would significantly alter the remainder of the analytical technique. Upon receipt of the data from CAL and the letter describing the data (January 21, 1986), CAL stated that "the dioxin and furan analyses were performed according to methods acceptable to EPA." No mention of EPA Method 8280 was made. Based upon prior knowledge of 8280 and a review of CAL's isomer class data, it was apparent that a method other than 8280 was performed. The comment must be made that Method 8280 has been under revision for several years and that the final validated 8280 method was not completed and released by EMSL-Las Vegas until March 1986. EG&G Idaho is obtaining a copy of Method 8280 from EMSL-Las Vegas for further review.

Upon review of this document, the use of multipoint calibration curves were confirmed, which CAL did not perform. Other differences in procedure were noted between the EPA Method 8280 method and the method CAL used. However, many of these differences are due to the multiple versions of 8280 in existence. The use of multipoint curves, however, has been mandatory in all versions.

The main concern is the use of single concentrations of analytical standards to determine response factors used in quantification calculations. Method 8280 stipulates multilevel calibration standards be used to determine response factors. This is an important consideration when a wide range of concentration values are anticipated, as was the case of the Huber samples.

As opposed to Method 8280, CAL ran a single point standard on a daily basis to determine response factors for the various analytical parameters. The standard was a mixture of polychlorinated dioxins and furans, which contained the following compounds:

CAL furnished the raw chromatograms of the standard analyses as well as their calculation sheets. These data were reviewed and calculations checked to verify numerical accuracy. Standards were either obtained commercially or manufactured by CAL. Sources of all standards were documented by CAL. The analyses were conducted using high-resolution gas chromatography/low-resolution mass spectrometry. The isomer class content analyses were conducted using a DB-5 fused silica capillary GC column 60 meters long. The temperature program used was to ramp the GC column temperature from 190°C to 305°C at a rate of 10°C/min.

The following ions were monitored to determine the presence of PCDDs and PCDFs as well as to provide information for quantification:

Compound	Nominal Mass 1	Nominal Mass 2	Theoretical Isotope Ratio Mass 1/Mass 2
TCDD	320	322	0.77
TCDD- <sup>13</sup> C <sub>12</sub>	332	334	0.77
P <sub>5</sub> CDD	354	356	0.617
$P_5^{CDD}$ - $\frac{13}{12}$	366	368	0.617
H <sub>x</sub> CDD	390	392	1.235
H_CDD- <sup>13</sup> C <sub>12</sub>	402	404	1.235
TCDF	304	306	0.77
TCDF- <sup>13</sup> C <sub>12</sub>	316	318	0.77
P <sup>2</sup> CDF	338	340	0.617
P <sub>3</sub> CDF = <sup>13</sup> C <sub>12</sub>	350	352	0.617
$_{\mathbf{x}}^{\mathbf{CDF}}$	372	374	0.514
$H_{\mathbf{x}}^{\text{CDF}}$ - $^{13}C_{12}$	386	390	2.858

The normally accepted practice is that the experimental isotope ratios should be within +15 percent of the theoretical value in order to be considered a positive indicator of the presence of a PCDD or PCDF. Upon reviewing CAL's data and documentation, it was hard to determine if this practice was followed for the isomer class data. CAL's supporting documentation focused primarily or 2,3,7,8-TCDD analysis, with little devoted to specific QA/QC criteria for the PCDD/PCDF analyses. Several Huber samples were determined to have internal standards with isotope ratios slightly outside the 15 percent boundary. As the number of analyses increases, it becomes more probable that some of the criteria will be marginal or slightly outside acceptable limits. Concern was exhibited only where exceptionally wide deviations from the 15 percent criteria were determined.

The following Huber samples were analyzed for total tetra through hexachlorinated PCDD and PCDF content:

HU-NCBC-R1-01	HU-NCBC-R1-02	HU-NCBC-R2-01
HU-NCBC-R2-02	HU-NCBC-R2-03	HU-NCBC-R1-09
HU-NCBC-R2-09	HU-NCBC-R2-09A	

In addition, a method blank and two native spikes were prepared. A duplicate sample using a Huber sample was not analyzed. The native spikes used samples HU-NCBC-R2-09A and HU-NCBC-R1-01.

The following samples were determined not to contain PCDDs of PCDFs at or above the reported detection limits: HU-NCBC-R1-02, HU-NCBC-R2-R2, HU-NCBC-R1-09, HU-NCBC-R2-09A, and the method blank. Sample HU-NCBC-R1-02, displayed a 1,2,3,4,7,8- $^{13}\mathrm{C}_{12}$ -H<sub>x</sub>CDF ratio of 3.389, which is slightly outside the 15 percent range. A more serious concern for this sample is that the M/2 404 ion for the H<sub>x</sub>CDF- $^{13}\mathrm{C}_{12}$  standard was not found due to interferences. This necessitated the calculation of the detection limit vs the TCDD- $^{13}\mathrm{C}_{12}$  internal standard. This sample had a total detection limit of 1.304 ppb, with fairly high detection limits for H<sub>x</sub>CDF, P<sub>5</sub>CDD, and H<sub>x</sub>CDD. Sample HU-NCBC-R2-02 displayed a H<sub>x</sub>CDD internal standard

ratio in excess of 15 percent. Samples HU-NCBC-R2-02 and HU-NCBC-R2-03 also displayed internal standard ratios outside 15 percent. None of the ratios was exceptionally far off.

The remainder of the Huber samples were determined to contain chlorinated dioxins or furans. When originally analyzed, sample HU-NCBC-R1-01 was determined to contain 118 ppb of tetra dioxin, with 115 ppb identified as 2,3,7,8-TCDD. This value did not agree with data gathered from isomer-specific data at other labs. CAL reran this sample under isomer-specific conditions and measured a 2,3,7,8-TCDD value of 193 ppb. CAL then scaled the total tetrachlorodibenzo-p-dioxin based on the 2,3,7,8-TCDD value in the following manner:

118/115 = 1.026 $1.026 \times 193 = 198.035 \text{ ppb}$ 

CAL attributed the differences in values to saturation of the mass spectrometer. However, no evidence of saturation is apparent from the CAL data. The discrepancy in values is probably because the initial analysis was conducted in the regime of nonlinear instrument response. The response of the instrument has reached a plateau and will not significantly change even with increasing concentration. For this type of situation, a multiconcentration calibration curve is essential. When CAL reanalyzed the sample using isomer-specific conditions, a smaller sample size was extracted and analyzed, thus introducing a smaller amount of material into the mass spectrometer. The same type of situation is present for HU-NCBC-R2-01.

Sample HU-NCBC-R2-03 was supplied with two data reports, one of which was a reexamination of the data using an enhanced software routine. Using the enhanced software, TCDF,  $P_5$ CDF, and TCDD were found. A maximum concentration of 0.49 ppb of 2,3,7,8,-TCDD was found. Upon isomer-specific analyses, it was determined that the 320/322 ratio was unacceptable (1.01), resulting in a maximum possible concentration of 0.78 ppb for 2,3,7,8-TCDD. Based on these conflicting results, a maximum possible concentration of 0.78 ppb may be present. For sample HU-NCBC-R2-03, the

calculations for  $H_X$  CDD could not be reproduced. The areas and heights used on the calculation sheets do not match those on the actual chromatograms. This is most likely the result of manually determining heights and areas and not noting them on the chromatogram.

Sample HU-NCBC-R2-02 was also supplied with two sets of data, one of which used the enhanced software processing. In the first set of data, results of "not detected" were reported for all isomer classes. Using the enhanced software, a value of 22 ppt (part per trillion) of TCDF was determined. Eased upon review of data, the positive identification of a TCDF is marginal because of signal-to-noise factors. The value of 28 ppt should be considered a maximum possible concentration.

As mentioned previously, two native spikes were prepared and analyzed. Of the two, the spike using the HU-NCBC-R2-09A sample was of better quality because the original sample contained no detectable amounts of dioxin and furans. Recovery of the native spikes ranged from 83 to 112 percent. All recoveries were well within the acceptable range. The spike sample using HU-NCBC-R1-01 provided good recoveries, with the exception of the TCDF and TCDD, because of the high levels of material already present in the sample.

In addition to the samples run by CAL for isomer class content, Battelle Columbus Laboratories analyzed sample HU-NCBC-R2-02 for tetra through hexa-chlorinated dioxins and furans using an in-house methodology (refer to Reference 2 for exact details). The results of this analysis show findings of "not detected" for all isomer classes, with detection limits ranging from 0.01 to 0.04 ppb. Battelle's data were examined and determined to met the QA/QC criteria described in the report.

Examination of CAL's isomer class data was a review and not a validation. When this work was performed, a single accepted, validated method for the isomer class determination was not available. Because of this, a uniform set of evaluation criteria has not been adopted. The review was aimed at understanding the data and analytical results and any isomossistencies noted. The data provided by CAL indicates trends in the

levels of PCDDs and PCDFs present and can be used on a semiquantitative basis to follow the destruction efficiency of the Huber process.

Because of the apparent switch by CAL from Method 8280 to what appears to be an in-house method, and the confused method of reporting, it was time-consuming and difficult to examine the data.

CAL also conducted 2,3,7,8-TCDD isomer specific analyses on specified samples and on those samples found to contain 2,3,7,8-TCDD when analyzed for isomer class content. CAL proposed to perform these analyses according to the U.S. EPA CLP method. According to the final report letter (January 21, 1986), this methodology was used with modifications made for extraction of various sample matrices. As stated previously, the data supplied by CAL were reviewed according to the same criteria used during the previous soil sampling and analysis program. Upon the completion of the review, it was apparent that the CLP procedure was not followed. Inconsistencies included the following:

- 1. No initial calibration curve established.
- 2. Incomplete concentration range of standards (100-ppb and 200-ppb standards omitted).
- 3. Partial scan was not provided until asked for.
- 4. Incomplete data reporting, including lack of initial calibration, lack of continuing calibration, lack of chronological list of all analyses performed.
- 5. Nonadherence to protocol concerning performance check standards.
- 6. Nonadherence to reporting format specified.

Because of these inconsistencies, the isomer-specific data for Huber can be technically considered invalid. The data can be used to project

trends in the Huber system's ability to decontaminate soil but would not be accepted by the Sample Management Office of the CLP.

In addition to the Huber samples, CAL analyzed a series of air filter samples using toluene Soxhlet extraction, followed by CAL's in-house 2,3,7,8-TCDD method. As before, these samples were to have been analyzed by the CLE method using suitable extraction modifications. The same deficiencies noted for the Huber 2,3,7,8-TCDD samples apply to the air filter samples.

Upon review of the data, samples 1138 and 1140 (which were reported to be 0.14 ppb and 0.11 ppb or 0.03 pg/m $^3$  and 0.07 pg/m $^3$ , respectively) should be changed to not detected, based on signal-to-noise criteria and have detection limits of less than 0.08 pg/m $^3$  and less than 0.18 pg/m $^3$ , respectively.

#### CONCLUSIONS

The CAL-supplied analytical data have numerous shortcoming and omissions that prevent strict validation of any of the results. However, the review/evaluation of the data has shown that the results can be used as indicative. Therefore, the results can be used to identify trends and to evaluate the probable effectiveness of the Huber process technology. The use of the results to provide strict quantitative information about the process is not justified without the additional corroborative information that would be provided by further testing of the Huber process.

The sample analyzed by Battelle was a sample of treated soil. As noted previously, it was found to be free of all isomer classes of both tetra through hexachlorinated dioxins and dibenzofurans down to detection levels that ranged from 0.01 to 0.04 ppb. The Battelle results were supported by adequate QA/QC and met all QA/QC criteria, as described in its report. Thus, these results are the most valid indication of the actual PCDD and PCDF levels in the treated soils. Since Battelle analyzed only a very limited number of samples, however, general conclusions based on its results must take that fact into account.

# REFERENCES

- 1. Review of Contractor Data from the IFB WA84-A002 Chemical Analytical Services for 2,3,7,8-Tetrachlorodibenzo-p-dioxin, Environmental Protection Agency, November 20, 1984.
- 2. Determination of Polychlorinated Dibenzo-p-dioxins and Poly-chlorinated Dibenzoturans in Soil Samples, Battelle Columbus Laboratories, Columbus, Ohio, May 21, 1986.

All 2,3,7,8-TCDD isomer-specific analytical data were reviewed and evaluated according to the requirements detailed in the EPA document for reviewing 2,3,7,8-TCDD analytical results (Reference 1 of Appendix 0). This document was adapted to form the working document used for detailed data review/evaluation. The criteria used to review the analytical data are as follows:

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- 1. To ensure isomer specificity for chromatographic separation, the 2,3,7,8-TCDD must be separated from interfering isomers with no more than a 50 percent valley relative to the 2,3,7,8-TCDD peak.
- 2. The m/z 320/322 and 332/334 ratios must be within the range of 0.67 to 0.87.
- 3. Ions 320, 322, and 257, which are each monitored separately but concurrently, must all be present; and the signal for all three must maximize simultaneously. The signal-to-noise ratio must be 2.5 to 1 or better for all three ions.
- 4. The signal-to-noise ratio must be 10 to 1 or better for the 332 and 334 ions, which are the ions due to the internal standard.
- 5. The retention time of the native 2,3,7,8-TCDD must equal (within 3 seconds) the retention time for the isotopically labeled 2,3,7,8-TCDD.
- 6. Positive results must be confirmed by obtaining partial scan spectra from mass 150 to mass 350 for selected samples.
- 7. The surrogate standard results must be within ±40 percent of the true value.

# APPENDIX R

# CALIFORNIA ANALYTICAL LABORATORIES PROTOCOL STANDARDS AND FOR ANALYSIS OF DIOXINS AND FURANS

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Exhibit	1	Analytical Methods - 2,3,7,8-TCDD in Soil and Sediment by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry	201
Exhibit	2	QA/QC Requirements for 2,3,7,8-TCDD Analysis	227
Exhibit	3	QA/QC Requirements for Dioxin and Furan Total Isomer Analysis	233
Exhibit	4	Sources of Standards and Internal Standards	234

# Exhibit D - Analytical Methods

2,3,7,8-tetrachlorodibenzo-p-dioxin in Soil 2,2, Sediment by High Resolution Gas Chromatographs Low Resolution Mass Spectrometry

#### 1. SCOPE AND APPLICATION

- 1.1 This method provides procedures for detection and measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD; CAS Registry Number 1746-01-6; STORET Number 34675) at concentrations of 1 µg/kg to 200 µg/kg in 10-g aliquots of wet soil and sediment. The use of 1-g aliquots permits measurement of concentration up to 2000 µg/kg.
- 1.2 The minimum measurable concentration is estimated to be 0.3  $\mu g/kg$ , but is dependent on interfering compounds present in the sample matrix.
- 1.3 This method is designed for use by analysts who are experienced in the use of a gas chromatograph/mass spectrometer.
- 1.4 CAUTION: Because 2, 3, 7, 8-TCDD is extremely toxic, safety procedures described in Section 5 of this method should be followed to prevent exposure of laboratory personnel to materials containing this compound.

#### 2. SUMMARY OF METHOD

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After 50 ng of  $^{13}\text{C-labeled}$  2,3,7,8-TCDD and 10 ng of  $^{37}\text{Cl-labeled}$  2,3,7,8-TCDD are added to a 10 gram aliquot of soil or sediment sample, the wet soil or sediment is mixed with 20 grams of anhydrous sodium sulfate an is extracted with a mixture of hexage and methanol, while the sample aliquot and solvent are agitated continually in a glass jar. Column chromatographic procedures are used to help eliminate sample components that may interfere with detection and measurement of 2,3,7,8-TCDD. The extract is concentrated to 50  $_{\text{U}}$ L, and a 2  $_{\text{U}}$ L aliquot is injected into a fused silica capillary column in a gas chromatograph (GC) interface to a mass spectrometer (MS) that has at least unit resolution at m/z 334.

Identification of 2,3,7,8-TCDD is based on detection of three characteristic ions, measurement of the appropriate relative abundances of two characteristic ions in the molecular ion cluster, and determination of the retention time of the sample analyte relative to the internal standard,  $^{13}C_{12}^{-2}$ ,3,7,8-TCDD, contained in the sample extract. The 2,3,7,8-TCDD concentration is determined by measuring the MS response to the sample component relative to the MS response to  $^{13}C_{12}^{-2}$ ,3,7,8-TCDD (the internal standard). The labeled internal

standard method presumes that internal standard losses during method procedures are equal to unlabeled TCDD losses. Therefore, the calculated sample 2,3,7,8,-TCDD concentration is corrected for losses during sample preparation.

The <sup>37</sup>Cl<sub>4</sub>-2,3,7,8- TCDD is a surrogate compound that is added to each sample and is analyzed exactly the same as unlabeled TCDD. The accuracy of surrogate compound measurement is used to indicate the accuracy of measurement of unlabeled 2,3,7,8-TCDD in the same sample.

#### 3. DEFINITIONS

- 3.1 Concentration calibration solution a solution containing known amounts of the analyte (unlabeled 2,3,7,8-TCDD), the surrogate compound (37Cl<sub>4</sub>-2,3,7,8-TCDD), and the internal standard (13C<sub>12</sub>-2,3,7,8-TCDD); it is used to determine instrument response of the analyte and the surrogate compound relative to the internal standard.
- 3.2 Field blank -- a portion of soil/sediment uncontaminated with 2,3,7,8-TCDD.
- 3.3 Rinsate -- a portion of solvent used to rinse sampling equipment and analyzed to demonstrate that samples are not contaminated during sampling.
- 3.4 Internal standard -- <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD, which is added to every sample and is present at the same concentration in every blank, quality control sample, and concentration calibration solution. It is added to the soil/sediment sample before extraction and is used to measure the concentrations of analyte and surrogate compound.
- 3.5 Laboratory reagent blank -- a blank prepared by the laboratory by performing all analytical procedures except addition of a sample aliquot to the extraction vessel.
- 3.6 Performance check mixture -- a mixture of known amounts of selected standard compounds; it is used to demonstrate continued acceptable performance of the GC/MS/DS system.
- 3.7 Performance evaluation sample -- a soil or sediment sample containing a known amount of unlabeled 2,3,7,8-TCDD. It is distributed by EPA to potential contractor laboratories who must analyze it and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). It may also be included as an unspecified QC sample in any sample batch submitted to the lab for analysis.

- 3.8 Response factor -- response of the mass spectrometer to a known amount of an analyte relative to a known amount of an internal standard.
- 3.9 Signal-to-noise ratio -- The ratio of the area of the analyte signal to the area of the random background signal; it is determined by integrating the signal for a characteristic ion in a region of the selected ion current profile where only random noise is observed and relating that area to the area measured for a positive response for the same ion. The same number of spectra must be integrated for both areas. (The ratio of peak heights may be used instead of peak areas.)
- 3.10 Surrogate compound -- <sup>37</sup>Cl<sub>4</sub>-2,3,7,8-TCDD, which is added to the soil/sediment before analysis. Its concentration is measured in each sample, and the accuracy of that concentration measurement is calculated to indicate the accuracy of the unlabeled 2,3,7,8-TCDD measurement.

#### INTERFERENCES

Any organic compound that is within 10 scans (at the rate of 1 scan/second) of m/z 257, 320, 322, or 328 of the internal standard and produces any of the three ions monitored to detect 2,3,7,8-TCDD, is a potential interference Most frequently encountered interferences are other sample components that are extracted along with TCDD. Because very low levels of TCDD must be measured, elimination of interference is essential. High purity reagents and solvents must be used and all equipment must be accupulously cleaned. Laboratory reagent blanks (Exhibit E, Quality Control, Section 4) must be analyzed to demonstrate lack of contamination that would interfere with TCDD measurement. Column chromatographic procedures are used to remove some coextracted sample components; these procedures must be performed carefully to minimize loss of TCDD during attempts to enrich its concentration relative to other sample components.

#### 5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a file of current OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are identified. (1-3) 2,3,7,8-TCDD has been identified as a suspected human or mammalian carcinogen.

- 5.2 Each laboratory must develop a strict safety program for handling 2,3,7,8-TCDD. The following laboratory practices are recommended:
  - 5.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.
  - 5.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or through a trap containing oil or high-boiling alcohols.
- 5.3 The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory are presented as guidelines only, and are based on safe handling practices included in USEPA Method 613. (4) The precautions for safe handling and use are necessarily general in nature because detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual usage. Assistance in evaluating the health hazards or particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. Although 2,3,7,8-TCDD is extremely toxic to laboratory animals, it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.
  - 5.3.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and lab hood adequate for radio-active work.
  - 5.3.2 Training: Workers must be trained in the proper method of removing of contaminated gloves and clothing without contacting the exterior surfaces.
  - 5.3.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift) with any mild soap and plenty of scrubbing action.
  - 5.3.4 Confinement: Isolated work area, posted with signs; segregated glassware and tools; and plastic-backed absorbent paper on benchtops.
  - 5.3.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

    Janiants should not handle wastes.
  - 5.3.6 Disposal of Wastes: 2,3,7,8-TCDD decomposes above 800°C.
    Low level waste, such as the absorbent paper and plastic gloves, may be burned in a good incinerator. Water containing gross quantities (milligrams) of 2,3,7,8-TCDD should be packaged securely and disposed through commercial or governmental channels that are capable of handling

high-level or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container; residues may then be handled as above.

- 5.3.7 Glassware, Tools, and Surfaces: Satisfactory cleaning may be accomplished by rinsing with 1,1,1-trichloroethane, then washing with any detergent and water. Dishwater may be disposed to the sever. (Also see Section 6.5.)
- 5.3.8 Laundry: Clothing known to be contaminated should be disposed with the precautions described under Section 5.3.6. Lab coats or other clothing worn in 2,3,7,8-TCDD work may be laundered. Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing. Disposable garments may be used to svoid a laundry problem, but they must be properly disposed or incinerated.
- 5.3.9 Wipe Tests: A useful method to determine cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, which is extracted and analyzed by gas chromatography (limit of sensitivity of approximately 0.1 µg per wipe). Less than 0.1 µg 2,3,7,8-TCDD per wipe indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 µg on a wipe sample indicates an acute hazard that requires prompt cleaning before further use of the equipment or work space and indicates that unacceptable work practices have been employed in the past.
- 5.3.10 Inhalation: Any procedure that may produce airborne contamination must be performed with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in case of an accident. Finely divided soils contaminated with 2,3,7,8-TCDD are hazardous because of the potential for inhalation. Such samples should be handled in a confined environment, such as a hood or glove box, or laboratory personnel should wear masks fitted with a particulate filter and charcoal sorbent.
- 5.3.11 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

#### 6. APPARATUS AND EQUIPMENT

- 6.1 Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS)
  - 6.1.1 The GC must be capable of temperature programming and be equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port must be designed for capillary columns. Splitless or on-column injection technique is recommended. With this method, a 2-\(\pu\)L injection volume is used consistently. With some GC injection ports, however, 1 \(\pu\)L may 1: the maximum volume that produces adequate precision and chromatographic separation. A 1-\(\pu\)L injection volume may be used if adequate sensitivity and precision can be achieved. CAUTION: If 1 \(\pu\)L is used for any injection volume, the injection volume for all extracts, blanks, calibration solutions and the performance check sample must be 1 \(\pu\)L.
  - 6.1.2 Mass spectral data are obtained with electron icnization at a nominal electron energy of 70 eV. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five data points for each of six ions while a sample component elutes from the GC.
  - 6.1.3 An interfaced data system (DS) is required to acquire, store, reduce and output mass spectral data. The DS must be equipped with a selected ion monitoring (SIM) program to acquire data for at least six ions that are characteristic of labeled and unlabeled 2,3,7,8-TCDD. (The mass spectrum of unlabeled 2,3,7,8-TCDD is shown in Figure 1 at the end of this Exhibit.) The same integration time must be used for each ion monitored, and the integration time used for sample analyses must be the same as the time used to analyze concentration calibration solutions and the performance check solution. Total data acquisition time per cycle (six ions) must not exceed 1.5 seconds.
- 6.2 GC Column -- Two fused silica capillary columns are recommended; one is a 60-m SP-2330 and the other is a 50-m CP-SIL 88. Any capillary column that separates 2,3,7,8-TCDD from all other TCDDs may be used, but this separation must be demonstrated. Minimum acceptance criteria must be determined per Section 9.2.3.1. At the beginning of each 8-hour period during which sample or concentration calibration solutions will be analyzed, column operating conditions must be demonstrated to achieve the required separation on the column to be used for samples. Operating conditions known to produce acceptable results with the recommended columns are shown in Table 1 at the end of this Exhibit.

# 6.3 Miscell neous Equipment

- 6.3.1 Nitrogen evaporation apparatus with variable flow rate from approximately 30 mL/min to 150 mL/min.
- 6.3.2 Mechanical shaker -- A magnetic stirrer or a wrist-action or platform-type shaker that produces vigorous agitation.

  Agitation conditions must be determined and demonstrated.
- 6.3.3 Analytical balance capable of accurately weighing 0.01g.
- 6.3.4 Centrifuge capable of operating at 2000 rpm.
- 6.3.5 Water bath -- equipped with concentric ring cover and temperature controlled within + 2°C.
- 5.3.6 Stainless steel spatulas or spoons.
- 6.3.7 Stainless steel (or glass) pan large enough to hold contents of 1-pint sample containers.
- 6.3.8 Glove box.

#### 6.4 Glassware

- 6.4.1 Extraction jars -- amber glass with Teflon-lined screw cap; minimum capacity of approximately 500 mL; must be compatible with mechanical shaker to be used.
- 6.4.2 Kuderna-Danish apparatus 500-mL evaporating flask, 10-mL graduated concentrator tubes with ground-glass stoppers, and 3-ball macro Snyder column.
- 6.4.3 Culture tubes -- 8-mL glass.
- 6.4.4 Mini-vials -- 1-mL amber borosilicate glass with conical-shaped reservoir and screw caps lined with Teflon-faced silicone disks.
- 6.4.5 Funnels -- glass; appropriate size to accommodate filter paper used to filter jar extract (volume of approximately 170 ml.).
- 6.4.6 Chromatography columns -- 1 cm ID x 10 cm long and 1 cm ID by 30 cm long.
- 6.5 NOTE: Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure.

Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of listilled water. Drain dry and heat in a muffle furnace at 400°C for 15 to 30 min. Volumetric glassware should not be heated in a muffle furnace, and some thermally stable materials (such as PCBs) may not be removed by heating in a muffle furnace. In these cases, rinsing with high-purity scetone and hexane may be substituted for muffle furnace heating. After glassware is dry and cool, store inverted or capped with aluminum foil in a clean environment.

#### / REAGENTS AND CONSUMABLE MATERIALS

- 7.1 Column Chromatography Reagents
  - 7.1.1 Alumina, acidic -- Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil covered glass container for 24 hours at 190°C.
  - 7.1.2 Silica gel -- high purity grade, type 60, 70-230 mesh; Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil-covered glass container for 24 hours at 130°C.
  - 7.1.3 Silica gel impregnated with 40% (by weight) sulfuric acid -Add two parts (by weight) concentrated sulfuric acid to
    three parts (by weight) silica gel (extracted and
    activated), mix with a glass rod until free of lumps, and
    store in a screw-capped glass bottle.
  - 7.1.4 Sulfuric acid, concentrated -- ACS grade, specific gravity 1.84.
  - 7.1.5 Graphitized carbon black (Carbopack C or equivalent), surface area of approximately 12 m<sup>2</sup>/g, 80/100 mesh.
  - 7.1.6 Celite 545R, reagent grade, or equivalent.
  - 7.2 Filter paper -- pore size of  $\leq$  20 to 25  $\mu$ ; rinse with hexane before use.
  - 7.3 Glass wool, silanized -- Extract with methylene chloride and hexane before use.
  - 7.4 Sodium sulfate -- Granular, anhydrous; before use, extract with methylene chloride and dry for > 4 h in a shallow tray placed in an oven operated at 120°C.

- 7.5 Solvents -- High puricy, distilled-in-glass; hexane, methanol, methylene chloride, and toluene.
- 7.6 Concentration Calibration Solutions (reference Table 2) -- Five toluene solutions containing unlabeled 2,3,7,8-TCDD at varying concentrations and \$^{13}C\_{12}-2,3,7,8-TCDD (the internal standard, CASRN 80494-19-5) at a constant concentration. Three of these solutions also contain \$^{37}Cl\_4-2,3,7,8-TCDD (the surrogate compound, CASRN 85508-50-5) at varying concentrations. Concentration calibration solutions are to be obtained from the Quality Assurance Division, USEPA Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. However, if not available from EMSL-LV, standards may be obtained from commercial sources, and solutions may be prepared in the contractor laboratory. Traceability of standards must be verified against EPA-supplied standard solutions, by laboratory SOP's as required in IFB Pre-Award Bid Confirmations, part 2.f.(4).
  - 7.6.1 Each of solutions #1-#5 contains \$\frac{13}{12}-2,3,7,8-TCDD at a concentration of 1 ng/\(\pi\)L which is equivalent to a 50-\(\pi\)L extract of a 10-g sample to which that compound (the internal standard) was added at a concentration of 5 \(\pi\)g/kg.
  - 7.6.2 Solutions #1-#5 contain unlabeled 2,3,7,8-TCDD at concentrations of 0.2, 1, 5, 20 and 40 ng/µL respectively; those concentrations are equivalent to 50-µL extracts of 10-g samples containing 1, 5, 25, 100 and 200 ppb, respectively.
  - 7.6.3 Solution #1-#3 contain <sup>37</sup>Cl<sub>4</sub>-2,3,7,8-TCDD at concentration of 0.06, 0.12, and 0.2 ng/<sub>µ</sub>L, respectively; those concentrations are equivalent to extracts containing 30, 60, and 100 ppb, respectively, of the amount of <sup>37</sup>Cl<sub>4</sub>-TCDD (the surrogate compound) added to each sample before extraction.
  - 7.6.4 Store concentration calibration solutions in 1-mL amber mini-vials at room temperature.
- 7.7 Performance Check Solution -- A mixture containing: unlabeled 2,3,7,8-TCDD; 1,2,3,4-TCDD (CASRN 30746-58-8); 1,4,7,8-TCDD (CASRN 40581-94-0); 1,2,3,7-TCDD (CASRN 67028-18-6); 1,2,3,8-TCDD (CASRN 53555-02-5); 1,2,7,8-(CASRN 34816-53-0) and 1,2,6,7-TCDD (CASRN 40581-90-6) must be obtained from the Ovality Assurance Division, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

To this dry mixture add 500  $_{\mu}L$  of the sample fortification solution (Section 7.8) containing  $^{13}C_{12}^{-2}$ ,3,7,8-TCDD at a concentration of 0.5  $n_{8}/_{\mu}L$  and  $^{37}C_{14}^{-2}$ ,3,7,8-TCDD at a concentration of 0.1  $n_{8}/_{\mu}L$ . Store in 1-pL amber mini-vial at room temperature.

- 7.8 Sample fortification Solution a toluran solution containing the internal standard at a concentration of 0.3 mg/g L and the surrogate compound at a concentration of 0.1 mg/g L.
- 7.9 Field Blank Fortification Solution a toluene solution -ontaining the internal standard at a concentration of 0.5 mg/<sub>k</sub> L, the surrogate compound at a concentration of 0.1 mg/<sub>k</sub> L, and the unlabeled 2,3,7,8-TCDD at a concentration of 0.1 mg/<sub>k</sub> L.

#### 8. SAMPLE PRESERVATION AND MANDLING

- 8.1 Chain-of-custody procedures -- see Exhibit G.
- 8.2 Sample Preservation

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- 8.2.1 When received, each sample will be contained in a 1-pint glass jar surrounded by vermiculite in a scaled metal paint can.

  Until a portion is to be removed for analysis, store the scaled paint cans in a locked limited-scaes area where ambient temperature is maintained between 0°C and 35°C.

  After a portion is removed for analysis, return the numsed portion of sample to its original containers and store as stated above. Do not freeze samples; they may contain sufficient water to break the sample jar if frozen.
- 8.2.2 To avoid photodecomposition, protect samples from light.

#### 8.3 Sample Handling

- 8.3.1 CAUTION: Finely divided soils contaminated with 2,3,7,8-TCDD are hazardous because of the potential for inhalation or ingestion of particles containing 2,3,7,8-TCDD. Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).
- 8.3.2 Pre-extraction sample treatment
  - 8.3.2.1 Homogenization -- Although sampling personnel will attempt to collect homogeneous samples, the contractor shall examine each sample and judge if it needs further mixing. NOTE: Contractor personnel have the responsibility to take a representative sample aliquot; this responsibility entails efforts to make the sample as homogeneous as possible. Stirring is recommended when possible.
  - 8.3.2.2 Centrifugation -- If a sample contains an obvious aqueous/liquid phase, centrifuge it to separate liquid and solid phases. Flace the entire sample in a suitable centrifuge bottle and centrifuge for 30

minutes at 2000 rpm. Remove bottle from centrifuge. With a disposable pipet, remove liquid phase and discard. CAUTION: This liquid may contain TCDD and should be disposed as a liquid waste. Mix solid layer with stainless steel spatula and remove a portion to be weighed and analyzed. Return the remaining solid portion to original sample bottle and store.

#### 9. CALIBRATION

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9.1 Two types of calibration procedures are required. One type, routine calibration, is required at the beginning and end of each 8-hour period during which TCDD analyses are performed. The other type, initial calibration, is required before any samples are analyzed for TCDD, and is required intermittently throughout sample analyses as dictated by results of routine calibration procedures described below. No samples are to be analyzed until acceptable calibration is demonstrated and documented.

#### 9.2 Routine Calibration

- 9.2.1 Calibrate and tune the MS with standards and procedures prescribed by the manufacturers. CAUTION: Some manufacturers may specify baseline resolution at masses higher than necessary for this method; that procedure could significantly reduce sensitivity for TCDD analysis.
- 9.2.2 Inject 2 pt (CAUTION: See Sect. 6.1.1) of the performance check solution (Sect. 7.7) and acquire selected-ion-monitoring mass spectral data for m/z 320, 322, 323, 328, 332, and 334 within a total cycle time of < 1.5 seconds. Acquire at least-five data points for each GC peak and use the same data acquisition time for each of the six ions being monitored. NOTE: The same data acquisition parameters previously used to analyze concentration calibration solutions during initial calibration must be used for the performance check solution.
- 9.2.3 Determine and document acceptable system performance with the following criteria:
  - 9.2.3.1 GC column performance -- If SP-2330 column is used, the valley between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers must be resolved with a valley ≤ 25%. Valley (%) = x/y % 100, when y is peak height of 2,3,7,8-TCDD; x is measured as shown in Figures 2 and 3 at the end of this Exhibit. The peak representing 2,3,7,8-TCDD shall be labeled and identified as such on the chromatograms.

- 9.2.3.2 Ratio of integrated ion current for m/z 320 to m/s 322 for 2,3,7,8-TCDD must be > 0.67 and < 0.87.
- 9.2.3.3 HS resolution -- Ratio of integrated ion current for m/z 323 relative to m/z 322 for unlabeled 2,3,7,8-TCDD should be > 0.07 and < 0.20.
- 9.2.3.4 Ratio of integrated ion current for m/z 332 to m/z 334 for  $^{13}C_{12}$ -2,3,7,8-TCDD must be  $\geq$  0.67 and  $\leq$  0.87.
- 9.2.3.5 Response factor (Sect. 9.3.10) for \$\frac{37}{Cl\_4} = 2,3,7,8 = TCDD relative to \$\frac{1}{3}C\_{12} = 2,3,7,8 = TCDD must be within + 10% of the mean value established by triplicate analyses of the concentration calibration solutions (Section 9.3).
- 9.2.4 Inject 2 µL of the concentration calibration solution #1, which contains 0.2 ng/µL of unlabeled 2,3,7,8-TCDD. Using the same GC/MS/DS conditions as used in Section 9.2.2 except the ions being monitored, acquire data for m/z 257, 320, 322, 328, 332, and 334. Determine and document acceptable performance for:
  - 9.2.4.1 MS sensitivity -- signal-to-noise (S/N) ratio (Section 3.8) of > 2.5 for m/z 257 and > 10 for m/z 322 for unlabeled 2,3,7,8-TCDD. The ratio of integrated ion current for m/z 257 to m/z 322 must be > 0.20 and < 0.45.
  - 9.2.4.2 Measured response factor for unlabeled 2,3,7,8-TCDD relative to \$^{13}C\_{12}-2,3,7,8-TCDD is within + 10% of the mean values established (Section 9.3) by triplicate analyses of the concentration calibration solutions.
- 9.2.5 Remedial actions shall be taken by Contractor if criteria are not met. Possible remedies are:
  - 9.2.5.1 Check and adjust GC and/or MS operating conditions.
  - 9.2.5.2 Replace GC column (performance of initial calibration procedures then required).
  - 9.2.5.3 Tune MS for greater or lesser resolution.
  - 9.2.5.4 Calibrate MS mass scale.
  - 9.2.5.5 Prepare and analyze new performance check solution.

# 9.2.5.6 Prepare new concentration calibration curve(s) (Section 9.3.11).

#### 9.3 Initial Calibration

- 9.3.1 In addition to routine calibration procedures described in Section 9.2, before any samples are analyzed, determine response factors for <sup>37</sup>Cl<sub>2</sub>-2,3,7,8-TCDD and for unlabeled 2,3,7,8-TCDD relative to <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD.
- 9.3.2 Concentration calibration solutions -- The five solutions described in Section 7.6 are required.
- 9.3.3 Calibrate and tune the MS with standards and procedures prescribed by the instrument manufacturer.
- 9.3.4 If a column other than the recommended (Section 6.2) SP-2330 or CP-SIL 88 fused silica capillary column is used, determine the GC conditions necessary to separate 2,3,7,8-TCDD from other TCDDs known to have similar relative retention times.
- 9.3.5 Inject a 2-µL aliquot of the performance check solution (CAUTION: See Section 6.1.1) and acquire selected-ion-monitoring (SIM) mass spectral data using the MS operating

conditions specified in Section 9.2.2. Determine GC operating conditions necessary to achieve separation described in Section 9.2.3.1.

- 9.3.6 Using specified MS data acquisition procedures and the GC conditions determined in Section 9.3.5, analyze a  $2-\mu L$  aliquot of the performance check solution.
- 9.3.7 Determine and document acceptable calibration using the criteria specified in Section 9.2.3.2 9.2.3.5.
- 9.3.8 Using the same GC conditions that produced acceptable results with the performance check solution, analyze a 2-µL aliquot of each of the five concentration calibration solutions with the following MS operating parameters.
  - 9.3.8.1 Acquire selected-ion-monitoring data for m/z 257, 320, 322, 328, 332 and 334.
  - 9.3.8.2 Total cycle time for data acquisition must be ≤ 1.5 seconds.
  - 9.3.8.3 Acquire at least five data points for each ion during elution of the GC peak.

- 9.3.8.4 Use the same data acquisition time for each of the six ions being monitored.
- 9.3.9 Repeat Section 9.3.8 two times to produce triplicate data sets for each solution.
- 9.3.10 Calculate the response factor for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and for unlabeled 2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD:

$$RF = \frac{A_x \cdot Q_{1q}}{A_{1s} \cdot Q_x}$$

where A = integrated ion abundance (corrected as specified in Section 12.1.1.3) of m/z 32d for 3/C1,-2,3,7,8,-TCDD or the sum of integrated ion abundances of m/z 320 and m/z 322 for unlabeled 2,3,7,8-TCDD,

A<sub>1s</sub> = the sum of integrated abundances of m/z 332 and m/z 334 for <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD,

 $Q_{is}$  = quantity of  $^{13}C_{12}$ -2,3,7,8-TCDD, and

Q = quantity of unlabeled 2,3,7,8-TCDD or  $\frac{37}{Cl_4-2}$ ,3,7,8-TCDD injected.

RF is a unitless number; units used to express quantities must be equivalent.

- 9.3.11 For both <sup>37</sup>Cl<sub>4</sub>-2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, calculate the mean RF and its relative standard deviation (RSD) from triplicate analyses of each of the five concentration calibration solutions. Variation of the RF calculated for each compound at each concentration level must not exceed 10% RSD. If the five mean RFs for each compound do not differ by more than ± 10%, the RF can be considered to be independent of analyte quantity for the calibration concentration range, and the mean of the five mean RFs shall be used for concentration calculations.
- 10. QUALITY CONTROL

See Exhibit E for QA/QC Requirements.

#### 11. PROCEDURES

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#### 11.1 Sample Extraction

- 11.1.1 CAUTION: See Section 5 for safety guidelines and recommendations.
- 11.1.2 Jar extraction. NOTE: Extremely wet samples may require centrifuging to remove water before addition of sodium sulfate see (Section 8.3.2.2).
  - 11.1.2.1 Accurately weigh to three significant figures a 10 gram (+ 0.5 gram) portion of the wet soil or sediment sample, and transfer it to the extraction jar.
  - 11.1.2.2 Add 100 pL of the sample fortification solution (Section 7.8) to the soil or sediment in the extraction jar. Add small portions of the solutions at several sites on the surface of the soil or sediment.
  - 11.1.2.3 Add 20 g of purified anhydrous sodium sulfate, and mix thoroughly using a stainless steel spoon or spatula.
  - 11.1.2.4 Allow the mixture of soil and sodium sulfate to set for two hours at embient temperature; mix again, break all visible lumps, and allow to set for at least four more hours.
  - 11.1.2.5 Mix again and add 20 mL of methanol; mix again and add 150 mL of hexane.
  - 11.1.2.6 Flace the extraction jar containing the soil, sodium sulfate and solvents in the shaker and shake for at least 3 hours.
  - 11.1.2.7 Remove the jar from the shaker and allow solids to settle. Decant the solvent through a glass funnel containing hexane-rinsed filter paper. Rinse the jar, solid sample residue, and filter residue with four 5-mL portions of hexane.
  - 11.1.2.8 Concentrate the extract volume to approximately 2 to 3 mL with a Kuderna-Danish apparatus or a rotary evaporator. NOTE: Glassware used for more than one sample must be carefully cleaned between samples to prevent cross contamination (See Section 6.5).

- 11.1.2.9 Transfer the concentrated extract to an 8-ul glass culturs tube. Rinse the evaporator flask with three 5-ul portions of hexane; transfer each rinse to the culture tube. Between additions of hexane rinse, reduce the extract, volume in the culture tube enough to allow addition of another 5-ul volume of rinse. To reduce the volume, place the culture tube in a water bath adjusted to operate at 50°C and position the tube so that the surfaces of the extract and the water are at about the same level. Evaporate the solvent with a stream of nitrogen (flow rate of approximately 150 mL/min) with the tip of the nitrogen delivery tube 2 cm above the solution.
- 11.1.2.10 After the final rinse has been added, reduce the extract volume to approximately 1 mL.

#### 11.2 Column Chromatography

#### 11.2.1 Column Preparation

- 11.2.1.1 Column 1: 21acs 1.0 g of silica gel into a 1 cm x
  20 cm column and tap the column gently to settle
  the silica gel. Add 2 g sodium hydroxideimpregnated
  silica gel, 1 g silica gel, 4.0 g of sulfuric acidimpregnated silica gel, and 2 g silica gel. Tap
  column gently after each addition.
- 11.2.1.2 Column 2: Place 6.0 g of alumina into a 1 cm x 30 cm column and tap the column gently to settle the alumina. Add a 1-cm layer of purified sodium sulfate to the top of the alumina.
- 11.2.1.3 Add hexand to each column until the packing is free of channels and air bubbles. A small positive pressure (5 psi) of clean nitrogen can be used if needed.
- 11.2.2 Quantitatively transfer the hexane sample extract from the culture tube to the top of the sulfuric acid-impregnated silics gel in Column 1. Rinse the culture tube with two 0.5 mL portions of hexane; transfer rinses to Column 1.
- 11.2.3 With 90 mL of hexane, elute the extract from Column 1 directly into Column 2 containing alvains and sodium sulfate.
- 11.2.4 Add 20 mL of hexane to Column 2 and elute until the hexane level is just below the top of the sodium sulfate; discard the eluted hexane.

- 11.2.5 Add 20 mL of 20% methylene chloride/80% hexche (volume/volume) to Column 2 and collect the eluste.
- 11.2.6 Reduce the volume of eluate with a gentle stream of filtered dry nitrogen. When the volume is about 1 to 2 mL, transfer aliquots to a 1-mL amber mini-vial with conical reservoir. Concentrate and add additional aliquots with further concentration until entire eluate is transfurred. Rinse eluate container with two 0.5-mL portions of hexane; transfer rinses to the mini-vial, with further concentration as necessary. CAUTION: Do not evaporate sample extract to dryness.
- 11.2.7 With the final sample extract volume at approximately 1 mL, store the extract until time for GC/MS analysis.

## 11.3 GC/MS Analysis

11.3.1 Remove the samp's extract or blank from storage and allow it to warm to ambient laboratory temperature if necessary.

With a stream of dry, filtered nitrogen, reduce the extract/blank volume to near dryness. Immediately before GC/MS analysis, adjust the extract or blank volume to 50 µL with toluene.

- 11.3.2 Inject a 2-uL aliquot of the extract into the GC, operated under conditions previously used (Sect. 9) to produce acceptable results with the performance check solution.
- 11.3.3 Acquire mass spectral data for the following selected characteristic ions: m/z 257, 320, and 322 for unlabeled 2,3,7,8-TCDD; m/z 328 for <sup>37</sup>Cl<sub>4</sub>-2,3,7,8-TCDD; and m/z 332 and 334 for <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD. Use the same data acquisition time and MS operating conditions previously used (Sect. 9.3.8) to determine response factors.
- 11.4 Identification Criteria. NOTE: Befor to Exhibit E, Section 7, for application of identification criteria.
  - 11.4.1 Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the  $^{13}C_{12}$ -2,3,7,8-TCDD. Recention times are required for all chromatograms, but scan numbers are optional. These parameters should be printed next to the appropriate peak.
  - 11.4.2 The integrated ion currents detected for m/z 257, 320, and 322 must maximize simultaneously. If there are peaks that will affect the maximization or quantitation of peaks of interest, attempts should be made to marrow the scan window to eliminate the interfering peaks. This should be reported on a separate chromatogram.

- 11.4.3 The integrated ion current for each analyte end surrogate compound ion (m/z 257, 320, 322 and 328) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background and must not have saturated the detector.
- 11.4.4 Relative abundance of m/s 257 to m/s 322 should be  $\geq$  20% and  $\leq$  45%.
- 11.4.5 Abundance of integrated ion counts detected for m/z 320 must be > 67% and < 87% of integrated ion counts detected for m/z 322.
- 11.5 Column Chromatography Procedure for Difficult Samples -- Use the following procedure for extracts previously subjected to the column chromatography procedures in Section 11.2, but found by GC/MS analysis to contain interfering components.
  - 11.5.1 Mix 3.6 grams of Carbopack C (or equivalent) with 16.4 grams of Celite 545 (or equivalent) in a 40-mL vial and activate by heating in an oven at 130°C for 6 hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopack/Celite to ensure TCDD recovery of > 50%. Subject the low level concentration calibration solution to this procedure and measure the quantity of isbeled and unlabeled 2,3,7,8-TCDD.
  - 11.5.2 Insert a small plug of glass wool into a disposable pipet approximately 15 cm long by 7 mm 0.D. Apply suction with a vacuum aspirator attached to the pointed end of the pipet, and add the Carbopack/Celite<sup>R</sup> mixture until a 2 cm column is obtained.
  - 11.5.3 Pre-eluce the column with:
    - 11.5.3.1 2 mL of toluene
    - 11.5.3.2 1 mL of a mixture of 75% (by volume) methylene chloride, 20% methanol and 5% benzene
    - 11.5.3.3 1 aL of 50% (by volume) cyclohexane and 50% methylene chloride
    - 11.5.3.4 2 mL of hexage
  - 11.5.4 While the column is still wet with hexane, add the sample extract. Elute the column with the following sequence of solvents and discard eluents.
    - 11.5.4.1 2 mL of hexage

- 11.5.4.2 1 mL of 50% (by vo, me) cyclohexane and 50% methylene chloride
- 11.5.4.3 1 mL of 75% (by volume) methylene chloride, 20% methanol and 5% benzene
- 11.5.5 Elute with 2 aL of toluene and collect the eluent, which contains the TCDD.
- 11.5.6 Store the sample extract until just before GC/MS analysis.

## 12. CALCULATIONS

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#### 12.1 Concentration

- 12.1.1 Concentration when a linear response factor was obtained:
  - 12.1.1.1 Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_x = \frac{A_x \cdot Q_{i_x}}{A_{ii} \cdot RF \cdot W}$$

where

- C<sub>x</sub> = 2,3,7,8-TCDD concentration in micrograms per kilogram
- Am the sum of integrated ion abundance detected for m/s 320 and 322
- Ais = the sum of integrated ion abundances detected for m/z 332 cnd 334 (characteristic ions of 13C<sub>12</sub>-2,3,7,8-TCDD, the internal standard)
- Q<sub>is</sub> = quantity (in nanograms) of <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD added to the sample before extraction
- RF = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to  $^{13}C_{12}$ -2,3,7,8-TCDD
- weight (in grams) of wet soil or sediment sample.

- 12.1.1.2 If the calculated concentration of unlabeled 2,3,7,8—TCDD exceeds 200 µ2/kg, which is the maximum concentration of the concentration calibration solutions, the linear range may have been exceeded, and a smaller aliquot of that sample must be analyzed. Accurately weigh to three significant figures a 1-g aliquot of the wet soil/sediment. Add 100 µL of the sample fortification solution (Section 7.8), just as for the larger sample aliquot. Extract and analyze.
- 12.1.1.3 Calculate the concentration of the surrogate compound,  $^{37}$ Cl<sub>4</sub>-2,3,7,8-TCDD, using the formula:

$$C_0 = \frac{A_0 \cdot Q_{10}}{A_{10} \cdot R^2 \cdot R}$$

- Cg = concentration (in micrograms per kilogram)
  of the surrogate compound
- A<sub>B</sub> = total integrated ion abundance of m/z 328 after correction for the contribution by unlabeled 2,3,7,8-TCDD (correction -- eubtract 0.9% of the total integrated ion abundance detected for m/z 322 in the same sample extract)
- A<sub>18</sub> = the sum of integrated ion abundances detected for m/s 332 and 334 (characteristic ions of <sup>13</sup>C<sub>1,2</sub>-2,3,7,8-TCDD, the internal standard)
- Q<sub>is</sub> = quantity (in nanograms) of <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD added to the sample before extraction
- RF calculated mean response factor for 3/Cl<sub>4</sub>-2,3,7,8-TCDD relative to 13C<sub>12</sub>-2,3,7,8-TCDD
- W = weight (in grams) of wet soil or sediment sample.
- 12.2 Accuracy -- Calculate the accuracy (A) of the measurement of surrogate, 37Cl<sub>4</sub>-2,3,7,8-TCDD, using the formula:

Surrogate amount measured (nanograms)
Percent Accuracy = 10 ng X 100

12.3 Estimated Detection Limit -- For samples in which no unlabeled
2,3,7,8-TCDD was detected, calculate the estimated minimum detectable
entration, which is the concentration required to produce a
signal with area (or peak height) of 2.5 times the background signal
area (or peak height). The background area is determined by integrating ion abundances for either m/z 320 or 322 in the appropriate
region of the SICP, multiplying that area by 2.5, and relating the
product area to an estimated concentration that would produce that
product area.

Use the formula:

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$$C_E = \frac{2.5 \cdot A_v \cdot Q_{i,a}}{A_{i,a} \cdot RF \cdot W}$$

where  $C_E$  = estimated concentration of unlabeled 2,3,7,8-TCDD required to produce  $A_r$ 

A = peak height or integrated ion abundance for either m/z 320 or 322 in the same group of  $\geq$  5 spectra used to measure  $A_{18}$ 

 $A_{18}$  = peak height or integrated ion abundance for the appropriate ion characteristic of the internal standard, m/z 332 when m/z 320 is used to determine  $A_{21}$ , and m/z 334 when m/z 322 is used to determine  $A_{22}$ 

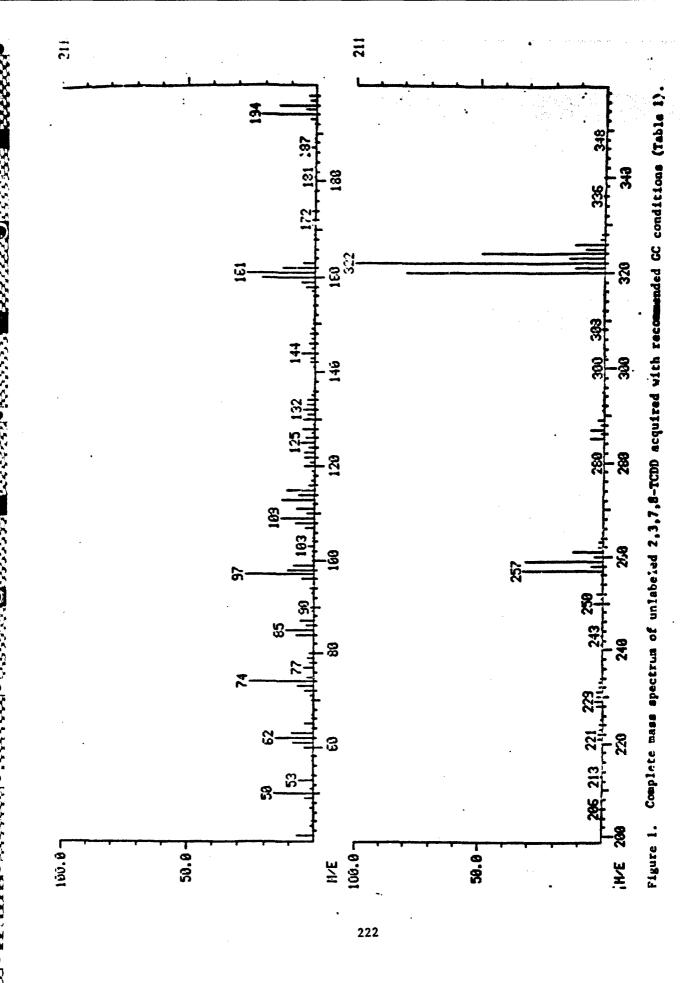
 $\mathbf{Q}_{\text{is}},~\mathbf{RF},~\mathbf{and}~\mathbf{W}$  retain the definitions previously stated in Section 12.1.1.

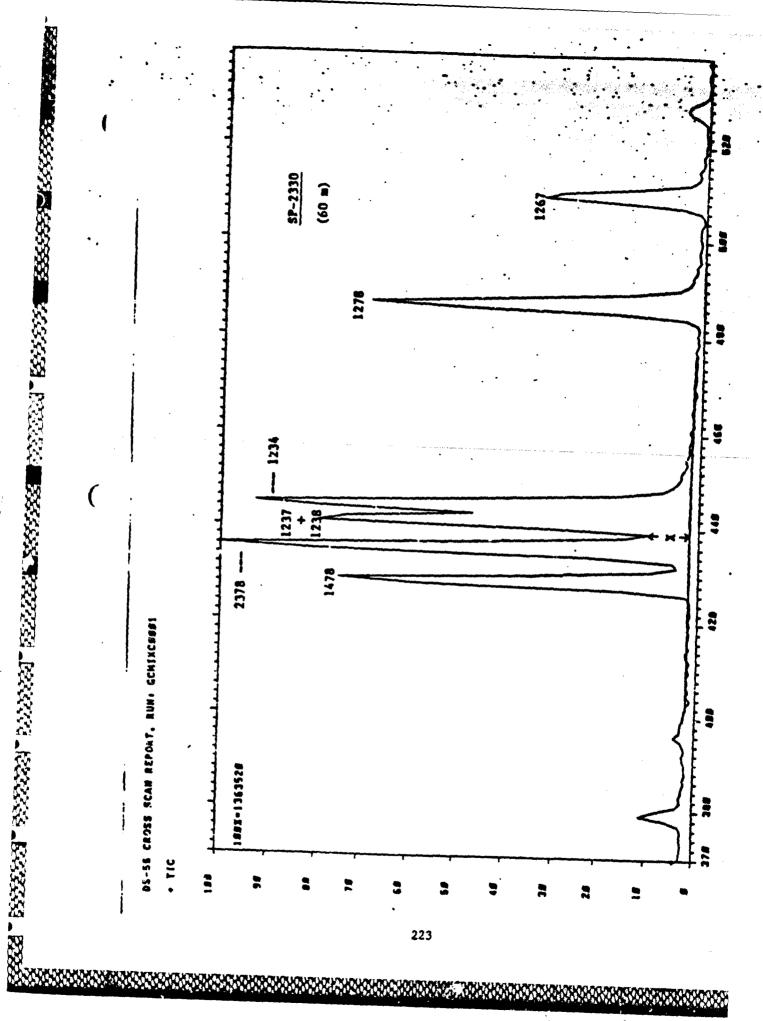
The use of the area (or peak height) for m/z 320 to calculate  $C_E$  is preferred to m/z 322, but m/z 322 can be used when interference is observed for m/z 320 but not for m/z 322.

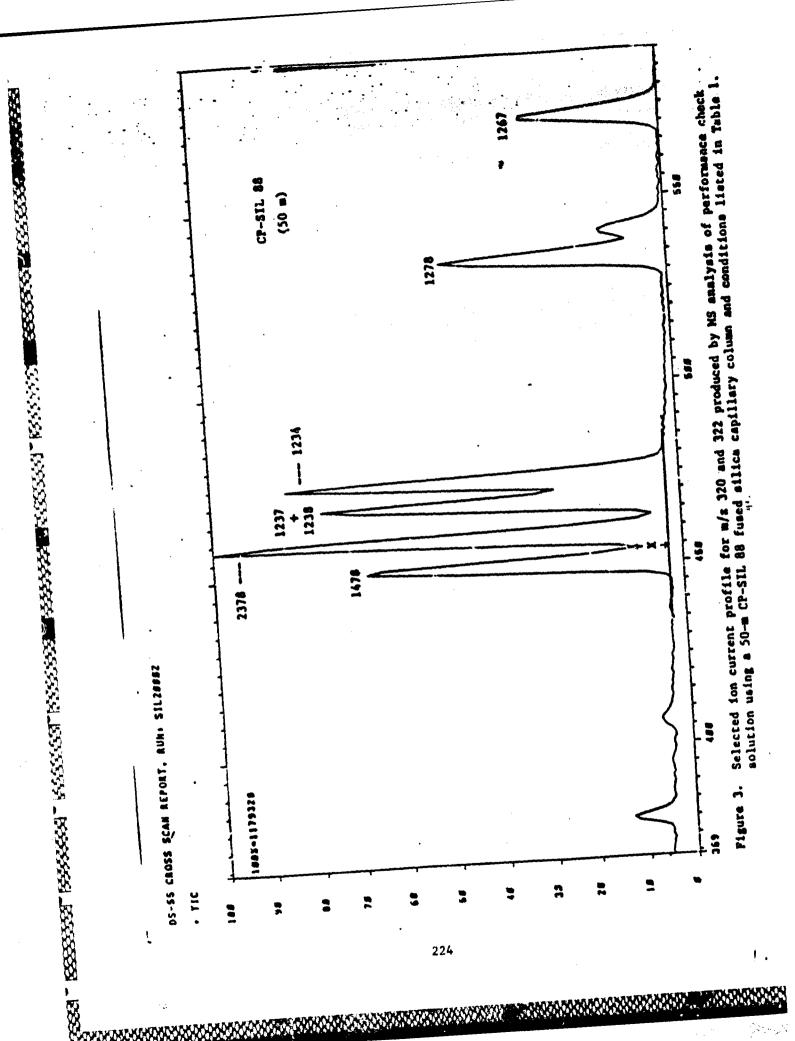
NOTE: This calculation is not applicable to all samples in which 2,3,7,8-TCDD was not identified (see Section 12.4).

- 12.4 Estimated Maximum Possible Concentration -- For samples where interference is observed for both m/z 320 and 322 or when an unacceptable ratio prevented identification of unlabeled 2,3,7,8-TCDD as a sample component, the procedure in Section 12.1 can be used to estimate the maximum concentration that could be represented by detected signals.
- 12.5 The relative percent difference (RPDZ) is calculated as follows: (See Section 5.1.1, Exhibit E.)

RPD = 
$$\frac{|S_1 - S_2|}{\text{Mean Concentration}}$$
 =  $\frac{|S_1 - S_2|}{\frac{|S_1 + S_2|}{2}}$ 







# TABLE 1. RECOMMENDED GC OPERATING CONDITIONS

Column coating	SP-2330	CP-SIL 88	
Film thickness	0. 2 <sub>µ</sub> m	0.22 <sub>µ</sub> m	
Column dimensions	60 m x 0.24 m1	50 m x 0-22 mm	
Helium* linear velocity	28-29 cm/sec at 260°C	28-29 cm/sec at 240°C	
Initial temperature	7 0°C	45°C	
Initial time	4 min	3 min	
Temperature program	Rapid increase to 200°C 200°C to 260°C at 4°C/min	Rapid increase to 190°C 190°C to 240°C at 5°C/min	
2,3,7,8-TCDD retention	24 min	26 min	

<sup>\*</sup> Rydrogen is an acceptable cerrier gas.

TABLE 2. COMPOSITION OF CONCENTRATION CALIBRATION SOLUTIONS

Solution #		Concentration of 2,3,7,8-TCDD	
	Isotopically Labeled		Unlabeled
	13 <sub>C12</sub>	37 <sub>C1</sub>	. •
1 2	1 ng/L 1 ng/L	0.06 ng/µL 0.12 ng/µL	0.2 ng/µL 1 ng/µL
3	1 ng/uL	0.2 ng/µL	5 ng/uL
• 4	1 ng/uL	0	20 ng/uL
5	l ng/"L	0	40 ng/,,L

# Exhibit E - QA/QC Requirements

#### SUMMARY OF OC ANALYSES

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- 1. Initial and periodic calibration and instrument performance checks.
- Laboratory reagent blank analyses (Sect. 4.1); minimum of one blank shall
  be analyzed with each sample batch; an additional blank analyzed when new
  reagents are used.
- 3. Analysis of a batch of samples with accompanying QC analyses:
  - 3.1 Sample Batch  $-- \le 24$  samples, including field blank and rineste sample(s).
  - 3.2 Additional QC Analyses Per Batch:

Laboratory reagent blank
Duplicate sample analysis
Confirmatory partial scan analysis
TOTAL
3

- 4. "Blind" QC samples may be submitted to contractor as an ordinary soil or sediment sample included among the batch of samples. Blind samples include:
  - 4.1 Uncontaminated soil,
  - 4.2 Split samples,
  - 4.3 Unlabeled duplicates, and
  - 4.4 Performance evaluation samples.

#### QUALITY CONTROL

- 1. Performance Evaluation Samples -- Included among samples in some batches will be samples containing known amounts of unlabeled 2,3,7,8-TCDD that may or may not be marked as other than ordinary samples.
- 2. Performance Check Solution
  - 2.1 At the begining of each 8-hour period during which samples are to be analyzed, an aliquot of the performance check solution and an aliquot of concentration calibration solution #1 shall be analyzed to demonstrate adequate GC and MS resolution and sensitivity, response factor reproducibility, and mass range calibration.

These procedures are described in Section 9 of Exhibit D. If any required criteria are not met, remedial action must be taken before any samples are analyzed.

- 2.2 To validate sample data, the performance check solution must be analyzed also at the end of each 8-hour period during which samples are analyzed.
  - 2.2.1 If the contractor laboratory operates only during one 8-hour period (shift) each day, the performance check solution must be analyzed twice (at the beginning and end of the 8-hour period) to validate data acquired during the interim period.
  - 2.2.2 If the contractor laboratory operates during consecutive 8-hour periods (shifts), analysis of the performance check solution at the beginning of each 8-hour period and at the end of the final 8-hour period is sufficient.
- 2.3 Results of at least two analyses of the performance check solution must be reported with sample data collected during an 8-h period.
- 2.4 Deviations from criteria specified for the performance check solution (Section 9.2.3, Exhibit D) invalidate all sample data collected between analyses of the performance check solution, and samples shall be rerun (see Exhibit C).
- 3. The performance check mixture, concentration calibration solutions, and the sample and field blank fortification solutions are to be obtained from EMSL-LV. However, if not available from EMSL-LV, standards can be obtained from other sources, and solutions can be prepared in the contractor laboratory. Concentrations of all solutions containing unlabeled 2,3,7,8-TCDD and not obtained from EMSL-LV must be verified by comparison to the unlabeled 2,3,7,8-TCDD standard solution (concentration of 7.87 µg/mL) that is available from EMSL-LV.

#### 4. Blanks

- 4.1 Laboratory reagent blank -- Perform all steps in the analytical procedure (Section 11, Exhibit D) using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis, but omit an aliquot of soil or sediment.
  - 4.1.1 Except in the case noted in Section 4.1.3, a laboratory reagent blank must contain the same amount of <sup>3/</sup>Cl<sub>4</sub>-2,3,7,8-TCDD and <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD that is added to samples before extraction.
  - 4.1.2 Analyze a laboratory reagent blank before any samples are extracted and analyzed.

- 4.7.3 Analyze two laboratory respent blanks before a new batch of solvents or reagents is used for sample extraction or for column chromatographic procedures. Do not add any \$\frac{3}{\text{Cl}\_4}=2,3,7,8=\text{TCDD or }\frac{1}{\text{Cl}\_2}=2,3,7,8=\text{TCDD to one blank, to demonstrate that reagents contain no impurities producing an ion current above the level of background noise for m/z 328, 332 and 334.
- 4.1.4 Analyze a laboratory reagent blank along with each batch of samples.
- 4.1.5 Acceptable laboratory reagent blanks contain no ion current above the level of background signal-to-noise for any of the selected characteristic ions (m/z 257, 320, 322) for unlabeled 2,3,7,8-TCDD. If the reagent blank which was extracted along with a batch of samples is contaminated, the entire batch of samples must be rerun (see Exhibit C).
  - 4.1.5.1 If the above criterion is not met, check solvents, reagents, apparatus, and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed.
  - 4.1.5.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.
- 4.2 Field Blanks -- Each batch of samples contains a sample of uncontaminated soil/sediment that is to be fortified with unlabeled 2,3,7,8-TCDD at a concentration of 1 µg/kg before analysis. In addition to that field blank, a batch of samples may include a rinsate, that is a portion of solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that samples have not been contaminated by sampling equipment.
  - 4.2.1 Unfortified field blank -- Analyze with procedures used for environmental samples (Section 11, Exhibit D). This blank may or may not be labeled as such (i.e., it may be a "blind" QC sample).
  - 4.2.2 Fortified (Spiked) Field Blank
    - 4.2.2.1 Weigh a 10-g aliquot of the specified field blank sample and add 100 µL of the solution containing 0,1 ng/µL of unlabeled 2,3,7,8-TCDD, 0,5 ng/µL of <sup>13</sup>C<sub>1,2</sub>-2,3,7,8-TCDD, and 0.1 ng/µL of <sup>37</sup>Cl<sub>4</sub>-2,3,7,8-TCDD. (Analysis before fortification is not required because this field blank is known not to contain a detectable concentration of unlabeled 2,3,7,8-TCDD.)

- 4.2.2.2 Extract with the jar procedure (Section 11.1.2, Exhibit D) and analyze a 2-LL aliquot.
- 4.2.2.3 Calculate concentration (Section 12.1, Exhibit D) of both <sup>3/</sup>Cl<sub>4</sub>-2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, and accuracy (Section 12.2, Exhibit D) of each measured concentration.
  - 4.2.2.3.1 If accuracy of measured concentration of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD is >  $\pm 40\text{Z}$ , discard the results and repeat the fortified field blank extraction and analysis with a second aliquot of the specified field blank sample (see Exhibit C).

#### 4.2.3 Rinsate Sample

- 4.2.3.1 To 2 100-mL aliquot of equipment rinse solvent (rinsate sample), add 100 μL of the solution containing 0.5 ng/μL of <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDD and 0.1 ng/μL solution of <sup>3/</sup>Cl<sub>4</sub>-2,3,7,8-TCDD.
- 4.2.3.2 Using a Kuderna-Danish apparatus or a rotary evaporator, concentrate the volume to approximately 5 mL.
- 4.2.3.3 Transfer the total 5-mL concentrate in 1-mL portions to a 1 mL-amber mini-vial, reducing volume as necessary with a gentle stream of dry nitrogen.
- 4.2.3.4 Rinse container with two 0.5 mL portions of hexane and transfer rinses to the 1-mL amber mini-vial.
- 4.2.3.5 Just before analysis, reduce volume to near dryness; make to final volume of 50  $\mu$ L with isooctane. (Column chromatography is not required.)
- 4.2.3.6 Analyze an aliquot with the same procedures used to analyze samples (Section 11, Exhibit D).

#### 5. Duplicate Analyses

- 5.1 Laboratory duplicates -- In each batch of samples, locate the sample specified for duplicate analyses and analyze a second 10-g sample aliquot.
  - 5.1.1 Results of laboratory duplicates must agree within 50% relative difference (difference expressed as percentage of the mean). If relative difference is > 50%, Contractor shall immediately contact the Sample Management Office for resolution of the problem. Report all results.

# 5.1.2 Recommended actions to help locate problem:

- 5.1.2.1 Analyze an aliquot of the performance check sample to verify satisfactory instrument performance (Section 9, Exhibit D.)
- 5.1.2.2 If possible, determine that no error was made while weighing sample aliquots.
- 5.1.2.3 Review analytical procedures with performing laboratory personnel.
- 6. Accuracy of Measured Concentration of \$\frac{37}{\text{Cl}\_4-2,3,7,8-TCDD}\$ -- For each sample and blank, calculate the percent accuracy (Section 12.2, Exhibit D) of the measured concentration of \$\frac{37}{\text{Cl}\_4-2,3,7,8-TCDD}\$. If percent accuracy is \$\frac{+40\text{X}}{\text{for a sample, analyze a second aliquot of that sample and report both results (see Exhibit C). NOTE: low or high accuracy for a blank does not require discarding sample data but indicates a potential problem with future sample data.

#### 7. Identification Criteria

- 7.1 If any of the four initial identification criteria (Sections 11.4.1 -11.4.4, Exhibit D) are not met, the sample is reported not to contain unlabeled 2,3,7,8-TCDD at the calculated detection limit (Section 12.3, Exhibit D).
- 7.2 When the four initial identification criteria are met, but the fifth criteria, the isotopic abundance ratio for m/z 320 and 322 (Section 11.4.5, Exhibit D) is not met, that sample is presumed to contain interfering contaminants. Contractor shall use the second column chromatography procedure (Section 11.5, Exhibit D) to remove interferences from the extract, and shall reanalyze the sample. (See Exhibit C.)
- 8. Blind QC Samples -- Included among soil and sediment samples may be QC samples that are not specified as such to the performing laboratory. Typos that may be included are:
  - 8.1 Uncontaminated soil.
    - 8.1.1 If a false positive is reported for this sample, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C).
  - 8.2 Split samples -- composited sample aliquots sent to more than one laboratory-
  - 8.3 Unlabeled field duplicates -- two sliquots of a composited sample.
  - 8.4 Performance evaluation sample -- soil/sediment sample containing a known amount of unlabeled 2,3,7,8-TCDD.

- 8.4.4 If the performance evaluation sample result falls outside the acceptance windows established by EPA, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C). NOTE: EPA acceptance windows are based on historical data results.
- 9. Confirmatory Partial Scan Analysis

RECEIPED HANKERS

9.1 From each sample batch, select the sample extract containing the highest concentration of unlabeled 2,3,7,8-TCDD and analyze an aliquot by GC/MS under the same GC conditions used previously but with the MS tuned and calibrated to acquire data for the mass range m/z 150 to m/z 350. (If no sample in a batch contains unlabeled 2,3,7,8-TCDD, no confirmatory analysis is required for that batch.) Required calibration criteria for decafluorotriphenylphosphina introduced through the GC column shall be:

<u>u/z</u>	Relative Intensity
51	30 - 60 percent of base peak
68	< 2 percent of $m/z = 69$
70	< 2 percent of $m/z = 69$
127	40 - 60 percent of base peak
197	< 1 percent of base peak
198	100 percent (base peak)
199	5 - 9 percent of base peak
275	10 - 30 percent of base yeak
365	> 1 percent of base peak
441	less than $m/z = 443$
442	> 40 percent of base peak
443	17 - 23 percent of $a/z = 442$

- 9.2 MS data acquisition requirements shall be:
  - 9.2.1 Cycle time < 1.5 seconds.
  - 9.2.2 Acquisition of  $\geq 5$  spectra during elution of 2,3,7,8-TCDD from the GC.
- 9.3 Subtract an appropriate background spectrum, and plot a spectrum of 2,3,7,8-TCDD after background subtraction. (The person responsible for MS data interpretation is responsible for demonstrating that the background spectrum selected for subtraction was an appropriate spectrum.) Provide a hard copy of the background spectrum, the TCDD spectrum before subtraction, and the TCDD spectrum after subtraction. The quality of the plotted spectrum will be affected by other sample components that have approximately the same GC retention time and will be highly variable. Desired spectral features are:

Ratio of m/z 320 to 322 = 0.77
Ratio of m/z 320 to 324 = 1.58
Ratio of m/z 257 to 322 = 0.32
Ratio of m/z 257 to 259 = 1.03
Ratio of m/z 194 to 196 = 1.54
m/z 160 and 161 = > 10% of m/z 322

Because  $^{13}$ C<sub>12</sub>-2,3,7,8-TCDD, the internal standard, is present in every sample and has essentially the same retention time as unlabeled 2,3,7,8-TCDD, the spectrum after background subtraction will represent a mixture. When  $^{13}$ C<sub>12</sub>-2,3,7,8-TCDD is present at a higher concentration than unlabeled 2,3,7,8-TCDD, the resultant spectrum (Figure E-1) must be normalized to m/z 372 to demonstrate desired spectral features.

- 10. Records At each contractor laboratory, records must be maintained on site for six months after contract completion to document the quality of all data generated during contract performance. Before any records are disposed, written concurrence of the Contracting Officer must be obtained.
- 11. Magnetic tapes containing all raw GC/MS data (including performance check solution, blanks, and concentration calibration solutions) must be delivered to EMSL-LV when sufficient data to fill or nearly fill a tape have been collected or when all samples are completed, depending on which event occurs first.
- 12. Unused portions of samples and sample extracts must be preserved for six months after sample receipt; appropriate samples may be selected by EPA personnel for further analyses.
- 13. Reuse of glassware is to be minimized to avoid the risk of using contaminated glassware.

#### LABORATORY EVALUATION PROCEDURES

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On a quarterly basis, the EPA Project Officer and/or designated representatives shall conduct an evaluation of the leboratory to ascertain that the laboratory is meeting contract requirements. This evaluation will consist (.: 1) laborator analysis of a performance evaluation sample, and 2) laboratory sits visit by EPA officials and/or representatives. The evaluation procedures will be similar, but may not be identical to the evaluation performed as part of the pre-award bidder evaluation. (See IFB Pre-Award Bid Confirmations section.)

### QA/QC Requirements for Dioxin and Furan Total Isomer Analysis

Our approach to QA/QC for the tatra-hexa furans and dioxins parallels closely that for 2,3,7,8-TCDD. Items 3 through 9 of the outline below point out additional items not specifically mentioned in 2,3,7,8-TCDD methods.

- 1. Materials Examined for Contamination
  - A. Prior to start of project
  - B. Along with each set of analyses
- 2. Traceable Standards
  - A. 2,3,7,8-TCDD traceable to EFA reference
  - B. Other dioxins and furans traceable to EPA
- 3. Internal Standards for EACH chlorination level
  - A. TCDD internal standards traceable to EPA
  - B. Others synthesized at CAL Lab and reference to Rappe's materials
- 4. Column Performance

- A. 2,3.7,8-TCDD resolution as per EPA requirements
- B. Resolution of 1,2,3,4,7,8 and 1,2,3,6,7,8-HxCDF
- 5. Documentation of Mass Spectrometer Resolution (Low or High)
  - A. Hardcopy of profile data
- 6. Cleanup column chromatography Performance
  - A. 1,3,6,8-TCDD to HxCDD recovery within 30% of Internal Standards
- 7. Duplicate Analyses
  - A. Duplicates within 30% for most congeners.
- 8. Spikes of Representative Tetra-Hexa Chlorodickins and Furans
  - A. Recovery should be within 30%
- 9. Detection Limit Calculated for each Chlorination Level in each Sample

Please note that duplicates and matrix spikes are regarded as bullable samples.

## APPENDIX D: SOURCES OF STANDARDS AND INTERNAL STANDARDS

### CAL LABS THRED DIBENZODIOXINS

Isomer	Source
2,7-DCDD	RFR Corp.
1,2,4-TrCDD	RFR Corp.
1,2,3,4-TCDD 1,3,6,8-TCDD 2,3,7,8-TCDD	Christoffer Rappe Christoffer Rappe Radian Corp, USEPA
1,2,3,7,8-PnCDD	KOR Isotopes
1,2,3,4,7,8-HxCDD	KOR Isotopes
1,2,3,4,6,7,8-HpCDD	KOR Isotopes
OCDD	Ultra Scientific

### CAL LABS OWNED INTERNAL STANDARDS

150mer	Source			
13C-2,3,7,8-TCDF	Cambridge Isotope Lab (CIL)			
13C-1,2,3,7,8-PnCDF 13C-2,3,4,7,8-PnCDF	CAL LAB Synthesized, CIL CAL LAB Synthesized, CIL			
13C-1,2,3,4,7,8-HxCDF	CAL LAB Synthesized			
13C-1,2,3,4,6,7,8-HpCDF 13C-1,2,3,4,7,8,9-HpCDF	CAL LAB Synthesized CAL LAB Synthesized			
13C-CCDF	CAL LAB Synthesized			
13C-2,3,7,8TCDD	KOP Istopes, USEPA			
13C-1,2,3,7,8-FnCDD	CAL LAB Synthesized, CIL			
13C-1,2,3,4,7,8-HxCDG	CAL LAB Synthesized, CIL			
13C-1,2,3,4,6,7,8-HpCDD	CAL LAB Synthesized			
13C-OCDD	KOR Istopes & CAL LAB Synthes			
Surrogate:				
37C1-2,3,7,8-TCDD	KOR Isotopes, USEPA			

### CAL LABS OWNED DIBENZOFURANS

Isomer	Source
2,8-DCDF	RFR Corp.
1,2,3,9-TCDF	Christoffer Rappe
1,2,4,7-TCDF	Christoffer Rappe
1,2,4,8-TCDF	Christoffer Rappe
1,2,6,7-TCDF	Christoffer Rappe
1,2,7,8-TCDF	Christoffer Rappe
1,2,7,9-TCDF	Christoffer Rappe
1,3,4,6-TCDF	Christoffer Rappe
1,3,6,7-TCDF	Christoffer Rappe
1,3,6,8-TCDF	Christoffer Rappe
1,3,7,9-TCDF	Christoffer Rappe
1,4,6,7-TCDF	Christoffer Rappe
1,4,6,9-TCDF	Christoffer Rappe
2,3,4,7-TCDF	Christoffer Rappe
2,3,4,8-TCDF	Christoffer Rappe
2,3,6,7-TCDF	Christoffer Rappe
2,3,6,8-TCDF	Christoffer Rappe
2,3,7,8-TCDF	Christoffer Rappe & Radian Corp.
2,4,6,7-TCDF	Christoffer Rappe
2,4,6,8-TCDF	Christoffer Rappe
1,2,3,4,8-PnCDF	Christoffer Rappe
1,2,3,7,8-PnCDF	Cambridge Isotope Lab
1,2,4,6,8-PnCDF	Christoffer Rappe
1,2,4,7,8-PnCDF	Christoffer Rappe
2,3,4,6,8-PnCDF	Christoffer Rappe
2,3,4,7,8-PnCDF	Christoffer Rappe
1,2,3,4,6,8-HxCDF	Christoffer Rappe
1,2,3,4,7,8-HxCDF	Cambridge Isotope Lab
1,2,3,4,7,9-HxCDF	Christoffer Rappe
1,2,4,6,7,8-HxCDF	Christoffer Rappe
1,2,4,6,8,9-HxCDF	Christoffer Rappe
2,3,4,6,7,8-HxCDF	Christoffer Rappe
1,2,3,4,6,7,8-HpCDF	Christoffer Rappe & Cambridge Isotopes
1,2,3,4,6,8,9-HpCDF	Christoffer Rappe
1,2,3,4,7,8,9-HpCDF	Christoffer Rappe
OCDF	Christoffer Rappe & Ultra Scientific

### APPENDIX S

# CALIFORNIA ANALYTICAL LABORATORIES DATA SHEETS FOR DIOXIN/FURAN ANALYSES, ORGANIC COMPOUND ANALYSES, AND INORGANIC ANALYSES

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Report Date: Column: SP-233

TCDD DATA REPORT

California Analytical Laboratories 2544 industrial Blvd. W. Secremento, CA 95691

Lab: California Analytical Laboratories Case No. 21932 Batch/Shipment No.

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		ž		1526898	3127880 134	222	\$55065@mat i.	2677002	014771	10000		255090Ret	2235380	1586130	2896000	2959730	3591470	1886030
		Ħ		1096630	2336860	245439	2730770	2239606	200000	2007	2302400	1873420	1684630	1173730	2247320	2279920	2749520	1448850
		328		938741	203667	203381	2315250	1952223	850411	4003	100/66	1603652	1444240	1022516	1863680	1891322	2419758	1231550
		22		• !	555546	•	12632	62752	•	7 2 2	000	•		•	•	4581	53943	•
		23		•	1556918	•	16597	157416	,	7647		1226	•	8708		11936	141300	•
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		Ë	14.50.m	17.20.00	20.02	00:00:71	17:58:00	18:15:00	18:59:00	19:28:00	20.07.00	20.50	30.47.02	20:04:02	20:51:13	21:36:00	00:21:22	22:55:00
		rate	00/25/85	00/25/85	20/27/00	20/27/40	04/52/82	02/52/82	09/55/85	09/25/85	09/25/85	00/25.85	20/36/00	20/22/40	50/52/60	53/53/60	59/57/60	69/G/A
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	Sample		METHOD BLANK Y	METH.BLKK NS Y	1131	1132	1111	7117	,	1 (6)	1136	1137 Y	1138 Y	1139	A 07:1	1161	A 1711	•
3	eqe :	2	21932MB	21932MBNS	21532-1	21932.2	21012.1	21017	21017	1.16413	21937-2	21937-3	21937-4	21937-5	21937-6	21937.7	21037-8	

FB = Field Blank
ND = Not Detected
DL = Detection Limit
XX = Re-extraction Partial Scan/Confirmatory Analysis

239

WB = Mative TCD Spike

D = Duplicate/Fortified Field Blank
RI = Re-injection

\*Corrected for contribution by native TCDD; U.9% of m/z 322 subtracted

Commuts:

Ratio Unacceptable - The sample shows for current for m/e 320 and m/e 322, but their ratio is unacceptable (outside the 9.67 - 6.87 window) for positive 2,3,7,8-1000 identification.

# California Analytical Laboratories, Inc. POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21591

CLIENT ID: HU-NCBC-R1-01

Date Analyzed: 8/30/85

Column: DB-

CAL ID: 21591-1

Weight: 10.06 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	16.3 3.7	- -
penta (12378) (23478)	20.7 0.21 0.11	- -
hexa (123478)	1.0 ND	0.084
DIOXINS	•	
tetra (total) (2378+1234)	196 A 193	<b>-</b>
penta (12378)	4.6 1.5	-
hexa (123478)	2.1 ND	0.21

\* Accuracy 3701-TCDD = 94%

A = Data taken from 2,3,7,8-TCDD specific analysis

ND = Not Detected

PREPARED BY: 1714

APPROVED BY:

2481

# California Analytical Laboratories, Inc. POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21413

HU-NCBC

CLIENT ID: R1-02

Date Analyzed: 10/16/85

Column: DB-

CAL ID: 21413-2RXRX\$

Weight: 10.15 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	ND ND	0.036 0.036
penta	ND	0.063
hexa	ND	0.15
DIOXINS		
tetra (total) (2378+1234)	ND ND	0.057 0.057
penta	ND	0.29
hexa	ND	0.24

\$ Accuracy 37Cl-TCDD = 54\$

ND = Not Detected

PREPARED BY:

APPROVED BY:

COH

name.

1/2/86

# California Analytical Laboratories, Inc.

## POLYCHLORINATED DIOXIN/FURAN ANALYSIS

### TICKET NO. 21591

R2-02

CLIENT ID: HV-NCBC-02

Date Analyzed: 10/16/85

Column: DB-

CAL ID: 21591-3RX\$

Weight: 10.00 q

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	0.028 0.028	-
penta	ND	0.019
hexa	ND	0.020
DIOXINS		
tetra (total) (2378+1234)	ND ND	0.03 <i>6</i> 0.03 <i>6</i>
penta	ND	0.18
hexa	ND	0.070

% Accuracy 37Cl-TCDD = 89%

ND = Not Detected

PREPARED BY:

APPROVED BY:

DATE.

# California Analytical Laboratories, Inc. POLYCHLORINATED DIOXIN/FURAN ANALYSIS TICKET NO. 21591

CLIENT ID: HV-NCBC-R2-03

Date Analyzed: 10/16/85

Column: DB-

CAL ID: 21591-04RX\$

Weight: 10.06 g

<b>FURANS</b>	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total)	0.54	-
penta (12378) (23478)	O.29 ND ND	0.033 0.033
hexa	E.~	0.074
DIOXINS		
tetra (total) (2378+1234)	0.87 0.49	
penta	ND	0.47
hexa	ND	0.26

**% Accuracy 37C1-TCDD = 101%** 

ND = Not Detected

PREPARED BY:

APPROVED BY:

DATE

243

# California Analytical Laboratories, Inc. POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO: 21413

CLIENT ID: HU-NCBC-R1-09

DATE ANALYZED: 9/17/85

CAL ID: 21413-3

WEIGHT: 10.05 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT
Total TCDF	ND	0.011
2,3,7,8-TCDF	ND	0.011
Total PCDF	ND	0.040
1,2,3,7,8-PCDF	ND	0.040
2,3,4,7,8-PCDF	ND	0.040
Total HCDF	ND	0.021
1,2,3,4,7,8-HCDF	ND	0.021
DIOXINS		
Total TCDD	ND	0.037
2,3,7,8-TCDD	ND	0.037
Total PCDD	ND	0.18
1,2,3,7,8-PCDD	ND	0.18
Total HCDD	ND	0.046
1,2,3,4,7,8-HCDD	ND	0.046

ND = Not Detected RX = Re-extraction

RI = Reinjection

PREPARED BY:

APPROVED BY:

1115

DATE

### Calliornia Analytical Laboratories, Inc.

### POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO: 21591

CLIENT ID: HU-NCBC-R2-09A (19:00)

DATE ANALYZED: 9/17/85

CAL ID: 21591-5

WEIGHT: 10.13 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
Total TCDF	ND	0.0034
2,3,7,8-TCDF	ND	0.0034
Total PCDF 1,2,3,7,8-PCDF 2,3,4,7,8-PCDF	ND ND	0.013 0.013 0.013
Total HCDF	ND	0.025
1,2,3,4,7,8-HCDF	ND	0.025
DIOXINS		
Total TCDD	ND	0.013
2,3,7,8-TCDD	ND	0.013
Total PCDD	ND	0.15
1,2,3,7,8-PCDD	ND	0.15
Total HCDD	ND	0.043
1,2,3,4,7,8-HCDD	ND	0.543

ND = Not Detected RX = Re-extraction

RI = Reinjection

PREPARED BY:

APPROVED BY:

DATE

10/22/55

## California Analytic: 1 Laboratories, Inc.

## POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET KO: 21591

mislisted

CLIENT ID: HU-NCBC-R2-09A (19:40)

DATE ANALYZED: 9/17/85

CAL ID: 21591-6

WEIGHT: 10.14 g

FURANS	AMOUNT FOUND (ng/g)	(ng/g)
Total TCDF	nd	0.0075
2,3,7,8-TCDF	nd	0.0075
Total PCDF	nd	0.021
1,2,3,7,8-PCDF	Nd	0.021
2,3,4,7,8-PCDF	Nd	0.021
Total HCDF 1,2,3,4,7,8-HCDF	ND ND	0.033 0.033
DIOXINS		
Total TCDD	ND	0.018
2,3,7,8-TCDD	ND	0.018
Total PCDD	ND	0.27
1,2,3,7,8-PCDD	ND	0.27
Total HCDD	ND	0.058
1,2,3,4,7,8-HCDD	ND	0.058

ND = Not Detected RX = Re-extraction

RI = Reinjection

PREPARED BY:

APPROVED BY:

266

### Organics Analysis Data Silest (Page 1)

Exhibit 3

Laboratory Name: <u>California /</u>	nalytical Laboratories, inc.	Case No: 21591	-	
Lab Sample ID No: 21591-1		OC Report No. NR	-	
Sample Matrix: SOIL		Contract No. NB.		
Data Release Authorized By: _	710	Date Samply Seceived: 6/29/85		
		ompounds		
	Concentration: Medium			
	Date Extracted/Prepared: 9/	16/85		
	Date Analyzed: 9/16/85			
	Conc/Dil Factor: 100 p	H:NR		
	Percent Moisture: NR	Principle - residue to the Mariana constitution resembles and the second second		
	Percent Moisture (Decanted	): <b>NR</b>		
CAS		CAS		

Number		ug/Kg
74-87-3	Chloromethene	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyi Chloride	209 U
75-00-3	Chloroethane	200 U
75-09-2	Methylane Chloride	\$00 U
67-64-1	Acetone	900 U
75-15-0	Carbon Disulfide	200 U
78-35-4	1,1-Dichleresthene	200 U
75-34-3	1,1-Dichloresthane	200 U
156-60-5	Trans-1,2-Dichlorosthene	200 U
67-06-3	Chloratorm	200 U
107-06-2	1,2-Dichloroethene	200 U
78-83-3	2-Butenone	800 U
71-55-6	1,1,1-Trichlorrethene	206 U
56-23-6	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetale	1000 U
75-27-4	Bromodichloromethene	200 U

Number		ug/Kg
76-87-6	1,2-Olchioropropene	200 U
10061-02-6	Trans-1,3-Dichloropropens	200 U
79-01-4	Trichi-roethene	200 U
124-48-1	Dibremochicremethene	200 U
79-00-6	1,1,2-Trichlerestinene	200 U
71-43-2	enectos	200 U
10061-01-8	cis-1,3-Dichleropropone	200 U
110-78-8	2-Chleroethylvinylether	1000 U
75-25-2	Bramatorm	200 U
108-10-1	4-Methyl-2-Pentanone	500 U
801-78-4	2-Hexanone	500 U
127-18-4	Tetraphierasthene	200 U
79-34-6	1,1,2,2-Tetrachloroethane	200 U
108-86-3	Teluene	200 U
108-99-7	Chicrober zene	200 U
100-41-4	Eihylbenzene	900 U
100-42-5	Styrene	200 U
	Total Xylensa	200 U

#### Data Reporting & saliflers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

Value If the result is a value greater than or equal to the detection limit, report the value.

- Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U Compound was analyzed for put not detected. The number is the minimum attainable detection limit for the name.
- Indicates an estimated value. This flag is used either when estimating a concentration for tentatively intentited compounds where a 1-1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection find but greater than zero. (e.g., 10J), if limit of detection is 10upf and a concentration of 3upf is calculated, report as 3J
- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS
- This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warms the data user to take appropriate action.

Other Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

NA Not Analyzed.
See cover letter.
Not Required.
Spikled Compound.

CLF. 11/14/85

rm i

Prepared by:

### **Organics Analysis Data Sheet** (Page 2)

### Serrivolatile Compounds

Concentration: MEDIUM	GPC Cleanup: NQ
Date Extracted/Prepared: 8/27/85, 4.3/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/12/85	Continuous Liquid - Liquid Extraction: NO

CAS Number 108-95-2 1.700 J bls(-2-Chioroethyl)Ether 111-44-4 4.X0 U 95-57-8 2-Cl-lorophenol 541-73-1 4679 U 1,3-Dichlorobenzene 105-46-7 4000 1,4-Dichlorobenzene 100-51-6 Benzyl Alcohol 4020 U

ConcrDiL Factor: 0.57G/ML

95-50-1 41/2 D U 1,2-Dichlorobenzene 4000 U 95-48-7 2-Methylphenicl 8000 U 39538-32-9 bis(2-chioroisopropyl)Ether 4-Methylphenol 4000 U 4000 U 621-64-7 N-Ntroen-Di-n-Propylamine 67-72-1 Hexachioro.ihane 4000 U 98-95-3 Nitrobenzene 78-59-1 laophorone 135-67-9 2,4-Dimethylphane 65-85-0 bls(-2-Chioroathory)Methane 111 91-1 120 83-2 2.4 Caltiforepaisonal 20-32-1 21-(7-3 Naphthelena 1 4/03 U 105-47-8 Herachio of Italiano 87 68-3 4-Chlura-2-Methy present 2-Metnylhaphthatene 91.57.6 Haxay - orda yellopynta flane 2,4,6-Trichlorophenol 88 06-2 95-95-4 2,4,5- inentorophenol 91-58-7 88-74-4 131-11-3

CAS		
Number		

Number		ug/Kg
83-32-9	Acenephthene	4000 U
51-28-5	2,4-Dinitrophenoi	2000ก บ
100-02-7	4-Nitrophenol	20000 U
132-64-9	Dibenzofuran	4000 U
121-14-2	2,4-Dinkrotoluene	8000 U
606-20-2	2,6-Dinitrotoluene	8000 U
84-65-2	Diethylphthalate	4000 U
7005-72-3	4-Chlorophenyl-phenylether	4000 U
86-73-7	Fluorene	4000 U
100-01-6	4-Nitroeniline	20000 U
534-52-1	4,5-Dinitro-2-Methylphenol	8000 U
86-30-6	N-Nit/oeodiphenylamine(1)	4000 U
101-55-3	4-Bromophenyl-phenylether	4000 U
118-74-1	Hexachiprobenzene	4000 U
87-86-8	Pentachlorophenol	4000 U
85-01-3	Phenanthrene	4000 U
120-12-7	Arithracene	4000 U
84-74-2	DI-n-Butylphthalate	4000 U
206-44-0	Fluorenthene	4000 U
129-00-0	Pyrene	4000 U
85-59-7	Butylbenzylphthalate	4000 U
91-94-1	3,3'-Dichlorobenzidine	8000 U
56-55-0	Benzo(a)Anthracene	4000 U
117-61-7	bie(2-Ethylhexyl)Phthalate	4000 U
218-01-9	Chrysene	8000 U
117-84-0	Di-n-Octyl Phthelate	4000 U
205-99-2	Benzo(b)Fluoranthene	8000 U
207-08-9	Benzo(k)Fluoranthene	8000 U
50-32-8	Benzo(a)Pyrene	8000 U
193-39-5	Indeno(1,2,3-cd)Pyrene	8000 U
53-70-3	Dibenz(a,h)Anthracene	8000 U
191-24-2	Benzo(g,h,l)Perylene	8000 U

(1) - Cannot be separated from diphenylamine

1000

CLF 10.1/85

208 96-8

99-09-2

Form I

### Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

Concentration: MEDIUM	GPC Cleanup: NO	
	Separatory Funnel Extraction: YES	
Date Extracted/Prepared: 8/27/85, 9/3/85	Septratory ("United Extractions, I had	
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO	
ConstDil Coston 0.11G/RMI		
Conc/Dil Factor: 0.11G/5ML		

CAS		
Number		ug/Kg
319-84-4	Alpha-BHC	80 U
319-86-7	Brite-BHC	<b>##</b> U
319-86-8	Delta-BHC	<b>80</b> U
54-00-0	Gemme-BHC (Lindane)	<b>80</b> U
78-44-8	Heptschier	<b>80</b> U
308-00-2	Aldrin	80 U
1024-47-3	Heptachier Epoxide	. 00 U
100-00-5	Enderuiten I	100 U
60-57-1	Dieldrin	100 U
73-55-0	4,4'-00€	100 U
72-20-4	Endrin	100 U
23213-66-0	Endersten II	100 U
72-84-8	4,4'-000	200 U
1031-87-8	Endocution Sulfete	200 U
80-20-3	4,4'-007	200 U
72-43-6	Metherychier	1800 U
83494-70-6	Endrin Ketone	NA
57-74-0	Chlordono	1000 U
8001-35-2	Temphone	10000 U
12674-11-2	Arestor-1016	NA.
11104-25-2	Arector-1221	NA
11141-16-6	Arester-1232	NA.
83469-21-8	Arecter-1242	1000 U
12672-28-6	Arester-1248	1000 U
11097-69-1	Arecier-1254	1006 U
11096-82-5	Arecter-1260	1000 U

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub>= Volume of water extracted (ml)

W<sub>a</sub>= Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

1008

Vs = NR

or W<sub>8</sub> =0.1

 $V_t = 5000$ 

V. = 5

249

CLF 11/14/85

Form I

manaday B

# Organics Analysis Data Sheet (Page 1)

	and the control of th
Laboratory Name: California Analytical Laboratories, Inc.	Case No: 21591
Lab Sample ID No: 21591-2	GC Report No: NR
Sample Matrix: SOIL	Contract No: NR
Data Release Authorized By:	Date Sample Received: 3/29/85

### Volatile Compounds

Concentration: 8" > A.1.
Date Extracted/Preparate, 2/16/85
Date Analyzed: 3/16/85
Conc/Dil Factor: 100 nH:NR
Percent Moisture: NR
Percent Moisture (Decanted): NR

#### CAS

Number		ug/Kg
74-87-3	Chioromethane	200 (1
74-83-0	Bromomethane	200 U
75-01-4	Ylnyt Chioride	200 tJ
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	500 U
87-64-1	Acetone	500 U
75-15-0	Carbon Disulficia	200 U
∷5-35-4	1,1-Dichioroethene	200 U
75-34-3	1,1-Dichloroethane	200 U
158-60-6	Trans-1,2-Dichro sethene	2000
67-66-3	Chloroform	j 206 U
107-06-2	1,2-Dichloroethane	200 U
78-93-3	2-Butanone	530 0
71-55-8	1,1,1-Trichtorcethe	:00 U
56-23-5	Carbon Tetrach Inde	250 U
108-05-4	Viny: Acetate	1700 U
75-27-4	Bromodichioromethane	ಜೂರ

### CAS

Number		ug/Kg
78-87-8	1,2-Dichloropropene	200 U
10061-02-6	Trans-1,3-Dichloropropens	200 U
79-01-6	Trichioroethene	200 U
124-48-1	Dibromochloromethene	2019 U
79-00-6	1,1,2-Trichlevoethene	200 U
71-43-2	Benzene	200 U
10061-01-6	de-1,3-Dichloropropene	200 U
£1 <b>9-75-8</b>	2-Chioroethylvinylether	1000 U
75-25-2	Eromotorm	200 U
106-10-1	4-Methyl-2-Pentanone	500 U
591-78-4	2-Kexanone	500 U
127-18-4	Tetrachloroethene	200 U
79-34-5	1,1,2,2-Tetrachioroethane	200 U
106-48-3	Toluene	200 U
108-80-7	Chiorobertzene	200 U
100-41-4	Ethylbenzene	500 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

### Data Reporting Qualifiers

For reparring results to  $\Gamma^{0}A$ , the following results qualifiers are used. Additional days or roots ofer explaining results are encouraged. However, the dot not of each flag must be explained.

Value If the result is a value greater than or equicito the detection cout, report the ketule

- Use Indicates compound wan sharyzed for human or derivated Report the man mumiders that arm tractifiers amplie with the U (e.g. 100) based on the restary concentration dilution achoes. (This is not no invisitely the instrument detection limit). The footnote should road U. Compound was unaryzed for but not detected. The number is the reinie unautal rate in telepoper.
- If indicates an extimation variety (i) is they all state over when estimating a love unmark. The device unless one dentified compounds where all the sponse is assumed or when the mass publication is not an entire drive of a compound that meets the interface of the form of the mass publication of the compound that meets the interface of the month of the compound that meets the interface of the month of the public is a finite or a state of the compounds of the co

- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS.
- This flag is used when the analyte is found in this blank as well as a sample. It indicates possible/probat a blank contamination and warms the data user to take appropriate action.

Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

NA Not Analyzed

NA Not Analyzed

See cover letter.

NR Not Required

Spiked Compound.

1039

CLF 11/14/85

Form I Prepa

Prepared by:

# Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

Concentration: Low	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/12/85	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 30G/10ML	,

CAS Number		ug/Kg
108-96-2	Phenol	3400
111-44-4	bie(-2-Chloroethyl)Ether	1000 U
95-87-8	2-Chlorophenal	1000 U
341-73-1	1,3-Dichlorobenzene	1000 U
106-46-7	1,4-Dichlorobengene	1000 U
100-51-6	Benzyl Alcohol	1000 U
95-50-1	1,2-Dic Marabenzene	1000 U
95-48-7	2-Methylphenol	1000 U
39638-32-9	ble(2-chlorolsopropyl)Ether	2000 U
106-44-5	4-Methylphenol	1000 U
621-64-7	N-Nitroso-Di-n-Propylamine	1000 U
67-72-1	Hexachloroethane	1000 U
94-95-3	Nitrobenzene	1000 U
78-59-1	leaphorone	1000 Ü
88-75-5	2-Nitrophenal	2000 U
105-67-9	2,4-Dimethylphenol	1000 U
65-85-0	Benzaic Acid	8000 U
111-91-1	bls(-2-Chloroethoxy)Methens	2000 U
120-83-2	2,4-Dichlorophunol	10000
.20-82-1	1,2,4-Trichiorobenzene	1000 U
91-20-3	Naphthalene	1000 U
106-47-8	4-Chioroeniline	1000 U
87-68-3	Hexachiorobutediene	1000 U
59-50-7	4-Chioro-3-Methylphenol	1000 U
91-57-6	2-Methylnaphthalene	1000 U
77-47-4	Mexachiorocyclopentadiene	1000 U
88-06-2	2,4,6-Trichlorophenol	55000 s
96-96-4	2,4,5-Trichlorophenal	
91-58-7	2-Chioronaphthaiene	1000 U
88-74-4	2-Nitroeniline	9006 U
131-11-3	Dimethyl Phthalate	1000 ij
208-96-8	Acenephthylene	1000 U
99-09-2	3-Mitroeniline	9000 U

CAS Number		ug/Kg
83-22-4	Aconophthone	1000 U
81-28-6	2,4-Dinkrophenol	9000 U
100-02-7	4-Nitrophenol	9000 U
132-64-0	Othenzofuren	1000 Ų
121-14-2	2,4-Dinkretoluene	2000 U
<b>COE-20-2</b>	2,6-Dinitrotaluene	2000 U
84-06-2	Disthylphthelate	1000 U
7005-72-3	4-Chlerophenyl-phenylether	1000 U
84-73-7	Fluerene	1000 U
100-01-6	4-Nitroeniline	9000 U
\$34-62-1	4,6-Dinitro-2-Methylphenol	2000 U
84-30-4	N-Mitrosodiphenylemine(1)	1060 U
101-65-3	4-Broms>honyl-phonylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
87-86-8	Pentachioraphenal	1000 U
85-01-8	Phononthrone	1000 U
120-12-7	Anthrecene	1000 U
84-74-2	Di-n-Butylphthelete	1008 U
206-44-0	Fluorenthene	1000 U
129-00-0	Pyrone	1000 U
85-68-7	Butyfeenzylphthelate	1000 U
91-84-1	3,3'-Dichiorobenzidine	2000 U
N-55-3	Benzo(s)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthelete	1000 U
218-01-8	Chrysene	2000 U
117-84-0	Di-ri-Octyl Phthelate	1000 U
205-09-2	Benzo(b)Fluorenthene	2000 U
207-08-9	Benzo(k)Fluoranthene	2000 U
50-32-4	Benzo(s)Pyrene	2000 U
193-39-6	Indeno(1,2,3-cd)Pyrene	2000 U
53-70-3	Dibenz(s,h)Anthracene	2000 U
191-24-2	Benzo(a.h.i)Perviene	2000 U

(1) - Cannot be secerated from dichenviamine

CLF: 10/11/85

Form I

repared by: \_\_\_\_\_\_

7/(

# Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

· • • · · · · · · · · · · · · · · · · ·	
Concentration: LOW	GPC Cleanup: NO
	Separatory Funnel Extraction: YES
Date Extracted/Prepared: 8/27/85	Separatory Further Extraction. 158
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 1.57G/50ML	

Number		ug/N
319-84-6	Alpha-BHC	30 U
319-85-7	Bets-BHC	30 U
319-86-4	Detta-BHC	30 U
58-89-9	Gamme-BHC (Lindene)	30 U
76-44-8	Heptachior	30 U
309-00-2	Aidrin	30 U
1024-57-3	Haptachlor Epoxide	30 U
959-98 8	Endosulfan I	ט סיל
60-57-1	Dieidrin	70 U
72-55-6	4,4'-DDE	70 U
72-20-8	Endrin	70 U
33213-65-9	Endoeultan II	70 U
72-54-8	4,4'-000	130 U
1031-07-8	Endosulfan Sulfate	130 U
50-29-3	4.4'-ODT	130 U
72-43-5	Hethoxychior	670 U
53494-70	g Endrin Ketone	NA
57-74-9	Chlordene	670 U
101-35-2	Toxaphene	6700 U
12674-11-2	Aroclor-1016	NA
11:04-28-2	Arocior-1221	NA
11141-16-5	Aroclor-1232	NA
53469-21-9	Aroclor-1242	670 U
12672-29-6	Aroclor-1248	670 U
11097-69-1	A:ocior-1254	670 U
11096-82-5	Aroclor-1260	670 U

V<sub>i</sub> = Volume of extract injected (ul)

 $V_{S}$  \* Volume of water extracted (ml)

W<sub>S</sub> = Weight of sample extracted (g)

 $V_t = Mc$  hime of total extract (ul)

 $V_{\rm S} = NR$  or  $W_{\rm S} = 1.57$ 

 $V_t = 50000$ 

1041

CLF 11/14/85

Form I

Prepared by:

### **Organics Analysis Data Sheet** (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.	Case No: 21413	
Lab Sample ID No: 21413-2	QC Report No: NR	
Sample Matrix: SOIL	Contract No: NR	
Data Release Authorized By:	Date Sample Received: 5/21/85	
	Compounds	
Concentration: Madium		
Date Extracted/Prepared:11	/14/85	
Date Analyzed: 11/14/85		
Conc/Dil Factor: 100	H:NR	
Percent Moisture: NR		
Percent Moisture (Decanted	d): NR	

CAS

Number		ug/Kg
74-87-3	Chloromethene	200 U
74-83-9	Sromomethane	200 U
75-01-4	Vinyl Chloride	200 U
78-00-3	Chloroethene	200 ປ
75-09-2	Methylene Chloride	800 U
67-64-1	Acetone	900 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichlorosthene	200 U
79-34-3	1,1-Dichloroethane	200 U
156-60-6	Trans-1,2-Dichioroethene	200 U
67- <del>66-3</del>	Chloreform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-93-3	2-Butanone	900 U
71-55 <del>-</del> 6	1,1,1-Trichlore-thane	200 U
56-23-5	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	, 1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number

ug/Kg 78-47-4 1,2-Dichloroprepene 200 U Trans-1,3-Dichloroprepene 10061-02-6 200 U 79-01-6 Trichlorosthens 200 U 124-48-1 Dibromochloromethens 200 U 79-00-8 1,1,2-Trichleroethene 200 U 71-41-2 200 U Bonzone 10061-01-5 cis-1,3-Dichleropropens 200 U 110-75-6 3-Chicrosthylvinylether 1000 U 75-25-2 206 U Brac storm 4-Methyl-2-Pentanone 500 U 591-78-6 2-Hexanone 900 U 127-18-4 Tetrachioroothene 200 U 1,1,2,2-Tetrachioroethane 73-34-6 200 U 106-80-7 Chlorobonzone 200 U 100-41-4 900 U Ethylbenstene 100-42-6 Styrene 200 U

#### **Data Reporting Qualifiers**

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

Value if the result is a value greater than or equal to the detriction limit, report the value.

- indicates compound was analyzed for but not desected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read; If-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the nemicle.
- Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence. of a compound that meets the identification crieris but the result is less than the specified detecton limit but greater than zero. (e.g. 10J), if limit of detecton is 10uo/1 and a concentration of 3ug/1 is calculated, report as 3J

This flag applies to posticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS.

Total Xylenee

This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warms the data user to take appropriats action.

Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data surmary report.

Not Analyzed.
See cover letter.
Not Required.
Spiked Compound.

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Form I

Prepared by: \_\_\_\_\_\_\_\_

10/85

CLF: 11/14/85

-	MALEGAL	
HU-NCE		

4 2-86

### Organics Analysis Data Sheet (Page 2)

#### Semivolatile Compounds

Concentration: MEDIUM	GPC Cleanup: NO
Date Extracted/Prepared: 9/3/85, 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 4/11/86	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 0.5a/0.5mi	

#### CAS Number ug/Kg 1000 U 108-95-2 111-4-4 ble(-2-Chloroethyl)Ether 1000 U 1000 U 95-57-8 2-Chlorophenol 1000 U 541-73-1 1,3-Dichlorobenzene 106-46-7 1,4-Dichlarobenzene 1000 U 1000 U 100-51-6 Benzył Alcohol 96-8C-1 1,2-Dichlorobergene 1000 U 1000 U 2-Methylphenol 95-48-7 2006 U 39638-32-9 bis(2-chloroisopropyi)Ether 106-44-5 4-Methylphenol 1000 U 1000 U 621-64-7 N-Nitrose-DI-n-Propylamine 1000 U 67-72-1 Hexaphioroethene 38-C8-3 Nitrobenzene 1000 U 1000 U 78-59-1 leophorone 2000 U 2-Nitrophenol 85-**73-5** 1000 U 105-57-9 2,4-Dimethylphenol 5000 U 55-85-0 Benzola Acid bis(-2-Chloroethoxy)Methane 2000 U 111-21-1 1000 U 140-83-2 2,4-Dichlarophenol 120-82-1 1,2,4-Trichlorobertzene 1000 U 81-20-3 1000 U Nephtheleno 1000 U 105-47-8 4-Chioronniline Hexachlorobutsdiene 1000 U 17-88-7 1000 U 56-70-7 4-Chloro-3-Methylphenol 2-Mathylnaphthalena 1000 U 21 57-6 1000 17 77-47-4 Hexachtorocyclopentadiene 1000 U 88-06-2 2,4,6-Trichlorophenai 95-95-4 2,4,5-Trichlorophenal 2000 U 1000 U 91-68-7 2-Chloronepmt:sleire 5000 U 55-74-4 2-Nitroeniline 1000 U 131-11-3 Dimethyl Phthalute 208-95-8 Acene phthylen-1050 U

CAS
Number

Number		ug/Kg
83.7.4	Acenephthene	1000 U
81-28-8	2,4-Dinkrophenol	50V6 U
100-02-7	4-Mitrophenol	5000 U
132-64-8	D'h-inzofureri	1060 U
121-14-2	2,4-Dinitrotoluene	2000 U
606-20-2	2,5-Dinitrataluene	2000 U
84-66-2	Diethylphtheiste	1000 U
7005-72-3	4-Chlorophenyl-phenylether	1000 U
84-73-7	Muorene	10/10 U
100-01-6	4-Mitrosniffine	3000 U
534-52-1	4,6-Dinitro-2-Methylphenol	2000 U
86-30-4	N-Nitrosodiphenylemins(1)	1000 U
101-68-3	4-Bramophenyl-phenylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
87-34-8	Pentachlorophenol	1000 U
85-01-8	Phenanthrene	1000 U
120-12-7	Anthracene	1000 U
84-74-2	Dt-n-Butylphthelate	1200
206-44-0	Fluoranthene	1000 U
129-00-0	Pyrene	1000 U
88-48-7	Sutylberizylphthelate	1000 U
91-94-1	3,3°-Dichlorobenzidine	2000 U
56-55-3	Benzo(a)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthalate	480 J
218-01-9	Chrysene	1000 U
117-84-0	DI-n-Octyl Philisiele	1000 U
205-09-2	Serrzo(b)Fluorenthene	1000 U
207-08-3	Benzo(k)Fluoremhene	1000 U
50-32-6	Bertzo(e)Pyrene	1000 U
123-35-1	Indeno(1,2,3-cd)Pyrene	1000 U
53-70-3	Ofbenz(a,h)Antiene	1000 U
191-24-2	Benzo(g,h,i)Perylene	1000 0

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Form I

5000 U

received by:

annot be separated from diphenylamine

7/85

CLF: 10/11/85

3-Mitroenfil-

99-09-2

71 F

# Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

Concentration: MEDIUM	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85, 9/13/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/16/85	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 0.10G/5ML	

Number		
18-84-4	Alphe-BHC	800
19-86-7	Bets-BHC	<b>89</b> U
19-86-8	Delta-SHC	80 U
8-19-0	Gemme-BFC (Lin Jenu)	80 U
<b>5-44-8</b>	Heptachier	80 U
09-00-2	Aldrin	80 U
024-57-3	Heptachior Epoxide	80 U
58-98-4	Endosuiten i	100 U
0-67-1	Dioldrin	100 U
2-46-0	4,4'-DOE	100 U
2-20-4	Endrin	100 U
3213-66-0	Endocultan II	100 U
2-64-6	4,4'-000	200 U
021-07-8	Endocution Sulfate	200 U
0-29-3	4,4'-007	200 U
2-43-6	Methoxychler	1000 U
3494-70-8	Endrin Katone	NA.
7-74-0	Chlordone	1000 U
001-35-2	Texaphene	10000 U
2674-11-2	Aracior-1016	NA
1104-28-2	Arecier-1221	NA.
1141-16-5	Arecter-1232	MA
3469-21-8	Alrector-1242	1000 U
2672-29-6	Arecier-1248	1000 U
1097-69-1	Arecier-1254	1000 U
1096-82-5	Arector-1250	1000 U

• V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub>= Volume of water extracted (ml)

W<sub>s</sub>= Weight of sample extracted (g)

V<sub>i</sub> = Volume of total extract (ul)

503

V<sub>S</sub> = NR

 $W_{\rm S} = 0.10$ 

 $V_{t} = 5000$ 

255

7/85

CLF: 11/14/85

10/85

# Organics Analysis Data Sheet (Page 1)

Laboratory Nan	ne: <u>California Analytical La</u> l	poratories, inc.	Case No: 21591		
Lab Sample ID	le ID No: 21591-3		QC Report No: NR		
Sample Matrix:			Contract No	· NR	
•	1)0	<u>1</u>	• • • • • • • •	e Received: <u>6/29/85</u>	
Data Helease A	Authorized By:		Date Sample	e neceived: 0/23/03	
		Volatile	Compounds		
	Concentra	ition: Med um			
	Date Extra	acted/Prepared:	9/16/85		
	Date Anal	yzed: 9/16/85			
	Conc/Dil F	actor: 100	pH:NR		
		oisture: NR			
	Percent M	oisture (Decante	ed): NH		
CAS			CAS		
Number		ug/Kg	Number		ug/Kg
74-87-3	Chloromethene	200 U	78-87-5	1,2-Dichtoropropane	500 U
74-83-9	Bromomethane	200 U	10061-02-6	Trans-1,3-Dichloropropene	200 U
75-01-4	Vinyl Chloride	200 U	79-01-6	Trichloroethene	200 U
75-00-3	Chloroethane Chlorida	200 U	124-48-1	Dibromochioromethane	200 U
75-09-2 67-64-1	Methylene Chloride	500 U	79-00-5	1,1,2-Trichioroethane	200 U
75-15-0	Acetone Carbon Disulfide	200 U	10061-01-6	Benzene	200 U
75-35-4	1,1-Dichloroethene	200 U	110-75-8	cis-1,3-Dichloropropene	200 U
75-34-3	1,1-Dichloroethane	200 U	75-25-2	2-Chloroethylvinylether  Bromoform	1000 U 200 U
156-60-8	Trans-1,2-Dichlorosthene	200 U	108-10-1	4-Methyl-2-Pentanone	200 U
67-66-3	Chloroform	200 U	591-78-6	2-Hexanone	500 U
107-06-2	1,2-Dichloroethana	200 U	127-18-4	Tetrachloroethene	200 U
78-93-3	2-Butanone	500 U	79-34-5	1,1,2,2-Tetrachloroethane	200 U
71-55-6	1,1,1-Trichioroethane	200 U	108-88-3	Toluene	200 U
56-23-8	Carbon Tetrachloride	200 U	108-90-7	Chiorobenzene	200 U
108-05-4	Vinyl Acetate	1000 U	100-41-4	Ethylbenzene	500 U
73-27-4	<b>Dromodichioromethane</b>	200 U	100-42-6	Styrene	200 U
				Total Xylenes	200 U
	Additional flag	esults to EPA, the folio	orting Qualiflers owing results qualifiers ( ing results are encourag	ere used. ed. Howevor, the	
Value If the result is a value greater than or equal to the detection limit, report the value  Undicates compound was analyzed for but not detected Report the minimum detection limit for the sample with			<b>c</b>	This flag applies to pesticide parameters identification has been confirmed by GC/I component pesticides >= 10 ng/ul in the fill should be confirmed by GC/MS	viS. Single
the U (e.g. 10U) based on necessary concentration/ dilution actions. (This is not necessarity the instrument detection limit.) The footnote should read. U Compound was analyzed for but not detected. The number is the minimum attainable detection limit for		B	This flag is used when the analyte is foun as well as a sample. It indicates possible blank contamination and warms the data u appropriate action.	/probable	
Indicates an estimated value. This flag is used either when estimated a concentration for tentativety identified compounds where a 1 1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10u). If limit of detection is 10up/1 2.5.6.  Other specific flags and footnotes may be reproperly define the results. If used, they mu described and such description attached to summary report.  NA Not Analyzed.  See cover letter.  NR Not Required.  Spiked Compound.		nust be fully			

Form I

CLF 11/14/85

4-22-86 submitted

8008 U 9000 U 1999 U 2000 U 2000 U 1000 U 1000 U 1000 U 9909 U 2008 U 129 J 1000 U 1909 U 1000 U 1009 U 1000 U 740 J 1000 U 1000 U 1000 U 2000 U 1000 U 1000 U 1000 U 1000 U 1000 U 1200 U 1000 U 1000 U 1000 U

# Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

CAS

Concentration: MEDIUM	GPC Cleanup: NO	
Date Extracted/Prepared: 8/27/85 & 9/3/85	Separatory Funnel Extraction: YES	
Date Analyzed: 4/11/85	Continuous Liquid - Liquid Extraction: MO	
Conc/Dil Factor: 0.51g/0.5ml		

CAS		
Number		ug/Kg
108-95-2	Phenol	1000 U
111-44-4	bis(-2-Chloraethyl)Ether	1000 U
15-57-8	2-Chlorophenol	1000 U
841-73-1	1,3-Dichierobengene	1000 U
105-46-7	1,4-Dichlerobenzene	1000 U
100-61-6	Sentyl Alcohel	1000 U
96-80-1	1,2-Dichlerobensone	1000 U
95-49-7	2-Mathylphenel	1005 U
39638-32-9	bis(2-chioroloograpyr)Ether	2000 U
108-44-8	4-Methylphenol	1000 U
621-44-7	N-Nitrose-Di-n-Propylamine	1000 U
67-72-1	Hexachieroethane	1000 Ų
96-95-3	Mitrobenzene	1000 U
78-99-1	lespherene	1000 U
88-75-6	2-Mitrophenal	2000 U
108-67-0	2,4-Olmothylphonol	1000 U
65-65-0	Benzole Acid	9000 U
111-01-1	bie(-2-Chioroethexy)Methene	2000 U
120-83-2	2,4-Cleidereghenel	1000 U
120-62-1	1,2,4-Trichlerebensone	1000 U
91-20-3	Haphthelana	1000 U
106-47-8	4-Chiercenffine	1000 U
87-68-3	Hexachlerobutadlene	1000 U
59-50-7	4-Chlore-3-Methylphenal	1006 U
91-57-4	2-Methylnephthelene	1006 U
77:47-4	Hexachiorocyclopentadiene	1006 U
80-06-2	2,4,6-Trichlorophenol	1000 U
16-16-4	2,4,5-Trichlorophenal	9000 U
91-58-7	2-Chloronophthelene	1000 U
86-74-4	2-Mitroenifine	8000 U
131-11-3	Directive Phthelete	1000 U
208-98-8	Acenephthylene	1000 U
99-09-2	3-Mitreenifing	9000 U

Number	
83-38-9	Assnaphthene
81-26-5	2,4-Dinitrophonol
100-02-7	4-Miraghanei
132-44-8	Otherspotunen
121-14-2	2,4-Dinitrotoksono
404-54-5	2.5-Dinitrateluene
94-95-2	Clathylphthelate
7006-773-3	4-Chlorophomyl-phomylether
<b>94-73-7</b>	Fluoron
100-01-6	4-Minoralling
<b>834-83-1</b>	4,6-Dinitre-2-Methylphonol
86-30-6	N-Mitrepediphenylainine(1)
101-65-3	4-Bromophomyl-phomylether
118-74-1	Homoshlorobongano
87-49-6	Pentgehlereghenel
85-91-6	Phononthrone
120-12-7	Arthresene
84-74-2	Cl-n-Butytehthelete
204-44-0	Researchers
120-00-0	Pyrane
<b>64.7</b>	Butythenzylphthelate
91-94-1	1.7-Olehlerebenzidine
PF-9F-3	Benze(e)Anthresene
117-81-7	bio(2-Ethylhoxyf)Phthalete
218-01-0	Chrysone
117-84-0	Di-n-Ootyl Phthelete
200-00-2	Benzo(b) Pluoranthone
297-00-0	Benze(t)/Rueranthene
50-32-4	Benze(s)Pyrene
193-39-6	Indena(1,2,3-cd)Pyrene
<b>83-70-4</b>	Ditions(s,h)Anthreeone
191-24-2	Benzo(g,h,f)Perylana

CLF: 10/11/85

1000 U

# Organics Analysis Data Sheet (Page 3)

### Pesticide/PCEs

Concentration: MEDIUM	GPC Cleanup: NO	
Date Extracted/Prepared: 8/27/85, 9/3/85	Separatory Funnel Extraction: YES	
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO	
Conc/Dil Factor: 0.11G/5ML		

CAS

Number		ug/K
319-84-6	Alpha-BHC	50 U
319-85-7	Beta-BHC	50 U
319-86-8	Defta-8HC	50 U
58-89-9	Gamme-BHC (Lindane)	50 U
76-44-8	Heptachlor	30 U
309-00-2	Aldrin	50 U
1024-67-3	Heptachlor Epoxide	50 U
959-98-4	Endosulfan i	106 U
60-57-1	Dieldrin	100 U
72-55-9	4,4'-DDE	100 U
72-20-8	Endrin	100 U
33213-65-9	Endosulfan II	100 U
72-54-8	4,4'-000	200 U
1031-07-8	Endosulfan Sulfate	200 U
50-29-3	4,4'-001	200 U
72-43-5	Methoxychior	1000 U
53494-70-5	Endrin Ketone	NA.
57-74-9	Chlordene	1000 U
8001-35-2	Toxaphene	10000 U
12674-11-2	Arocior-10100	NA
11104-28-2	Arocior-1221	NA
11141-100-5	Arocior-1232	NA
53469-21-9	Aroclor-1242	1006 U
12672-29-6	Arocio:-1248	1000 U
11097-69-1	Arucior-1254	1000 U
11096-82-5	Arocior-1260	1000 ປ

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>S</sub>= Volume of water extracted (ml)

W<sub>s</sub>= Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

1070

V<sub>s</sub> = NR

or  $W_S = 0.11$ 

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Form !

7/85

CLF 11/14/85

### **Organics Analysis Data Sheet** (Page 1)

Laboratory Name: California A Lab Sample ID No: 21591-4 Sample Matrix: SOIL Data Release Authorized By:	<u> </u>	Case No: 21581  QC Report No: NR  Contract No: NR  Date Sample Received: 5/29/85	
	Volatile C	ompound <b>s</b>	•
	Concentration: Medium		
	Date Extracted/Prepared: 9/	15/85	
	Date Analyzed: 9/16/85		
	Conc/Dil Factor: 100 p	H:NR	•
	Percent Moisture: NR		
	Percent Moisture (Decanted	: NR	
CAS		CAS	

Number

75-27-4

140111061		Ugrkg
"4 87-3	Chloromethane	200 U
74-83-8	Bromomethene	, 200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chiaride	800 U
67-64-1	Acetone	800 U
75-15-0	Carbon Disuifide	200 U
75-35-4	1,1-Dichloroethene	200 U
75-34-3	1,1-Dichloroethene	200 U
156-60-5	Trans-1,2-Dichlorosthens	200 U
67-66-3	Chloreform	200 U
107-06-2	1,2-Dichlorosthene	200 U
78-93-3	2-Butanone	800 U
71-55-6	1,1,1-Trichloroethane	200 U
54-23-5	Carbon Tetranioride	200 U
108-05-4	Vinyl Acetate	1000 U
	<del></del>	

Number ug/Kg 78-87-8 1,2-Dichioropropene 200 U 10061-02-6 Trans-1,3-Dichlorepropens 200 U 78-01-6 Trichioreethene 200 U 124-48-1 Dibremechloromethane 200 U 1,1,2-Triehloreethene 200 U 71-43-2 160 J 10061-01-5 sis-1,3-Dichleropropens 200 U 110-78-8 1000 U 2-Chloroethylvinylether /3-25-2 200 U 106-10-1 900 U 4-Methyl-2-Pentanone 501-78-6 3-Hexanone #00 U 127-18-4 Totrachieraethene 200 U 79-34-6 1,1,2,2-Tetrachleroethane 200 U 200 U 108-80-7 200 U 100-41-4 886 U Ethylbenzene 100-42-4 200 U Styrene **Total Xylenes** 200 U

### **Data Reporting Qualiflers**

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

200 U

Value If the result is a value greater than or equal to the detection limit, report the value.

Bromodichioromethane

- Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:7 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteris but the result is less than the specified detection kinst but greater than zero. (e.g. 10J). If limit of detection is 10ugl and a concentration of Sugf is calculated, report as 3J

- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS
- This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/pmbable blank contamination and warns the data user to take appropriate action.

Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report. Not Analyzed.

See cover letter.
Not Required.

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Form I

10/85

CLF: 11/14/85

4-22 20 successful

### Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

Concentration: MEDIUM	GPC Cleanup: NO	
Date Extracted/Prepared: 2/27/85 & 9/3/85	Separatory Funnel Extraction: YES	
Date Analyzed: 4/11/86	Continuous Liquid - Liquid Extraction: NO	
Conc/DiL Factor: 0.5g/0.5ml		

CAS

CLF: 10/11/85

Number		ug/Kg
108-95-2	Phenoi	1000 U
111-44-4	ble(-2-Chloroethyl)Ether	עי 1000
70-67-	2-Chlorophenol	1000 U
641- <b>73-1</b>	1,3-DiaNarabenzene	1006 U
108-46-7	1,4-Dichlarobergene	1000 U
100-51-4	Sanzyi Alsohol	1000 U
96-60-1	1,2-Dighiaroberzene	1000 U
rs-48-7	2-Methylphenol	1001 U
.9638-32-0	ble(2-chloro/sopropyl)Ether	2000 U
108-44-5	4-Methylphenol	1000 U
621-64-7	N-Hitroso-Ol-n-Propylamine	1000 U
o7-72-1	Hexachiorosthane	1000 U
02.04-3	Hitrobenzene	1000 U
77 <b>59-1</b>	Inophorone	1000 U
-8-79-5	2-Nitrophenol	2000 U
108-67-9	2,4-Olmethylphenal	1000 U
5-85-0	Benzola Acid	5000 U
111-01-1	ble(-2-Chloraethoxy)Methene	2000 U
120-25-2	2,4-Dichloroghunol	1000 U
·^n-82-1	1,2,4-Trichterebenzene	1000 U
31-20-1	Nephtralene	18000
1/23-47-8	4-Chierosnitine	1000 U
27-58-3	Hexachlorobutadiene	1000 U
50 6-L7	4-Chlore-3-Methylphenol	1000 U
-5 <u>18 </u>	2-Methylnaphthalene	1000 U
17 27-4	Hexachlorocyclopentediene	1000 U
88-08-2	2,4,8-Trichlarophenal	1000 U
25-05-4	2,4,5-Trichiorophenoi	5000 U
21-73-7	2-Chioronaprithalens	10°'-U
88-74	2-Nitrosniline	5000 U
131-11-3	Dimethyl Phthelate	1000 U
208-96-8	Acenephthylene	36300
99-09-2	3-Nitroeniline	8000 U

CAS

Number		ug/Kg
D-22-4	Accrephthene	1900
51-28-6	2,4-Oinitrophs.no.	5000 U
100-02-7	4-Mitrophenol	9600 U
132-44-9	Ditenzoluren	720 J
121-14-2	2,4-Dinkrototuene	29 <b>00 U</b>
604-20-2	2,5-Dinitrotoluene	2000 U
84-66-2	Diethylphthalate	1000 U
7006-72-3	4-Chlorophenyl-phen-fether	1000 U
86-73-7	Fluorene	1300
100-01-6	4-Nitroeniline	5000 U
534-62-1	4,5-Dinkro-2-Methylphers I	2000 U
86-30-6	N-Nitrosodiphenylamin./1)	) 40 J
101-65-3	4-Bromophernyl-phenylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
87-86-6	Pentachiorophenol	1000 U
85-01-4	Phenanthrene	13000
120-12-7	Anthrecene	2000
84-74-2	Cri-n-Buty/phthelate	1200
208-44-0	Fluorenthene	23000
129-00-0	Pyrone	47000
85-48-7	Butyfi enzylphthelate	1000 U
91-84-1	1,3'-Dichioroberzidine	2000 U
54-55-3	Benzo(s) Anthrecone	1000 U
117-81-7	bie(2-Ethylhexyl)Pithainte	800 J
218-01-9	Chrysene	1000 U
117-84-0	Di-n-Octyl Phthalate	1000 U
206-99-2	Benzo(b)Fluorenthene	1700
207-08-9	Benzo(k) Muoranthene	1700
50-12-8	Benzo(a)Pyrene	1000 U
195-39-6	Indenc(1,2,3-cd)Pyrane	. 1090 U
52.73.3	Dibenz(s,h)Anthrecene	1000 U
191-24-2	Senzo(g,h,l)Perylene	:000 U

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Form i

PIA

# Organics Analysis Data Sheet (Page 3)

#### Pesticide/PCBs

Concentration: MEDIUM	GPC Cleanup: NY 2
	Concessor Fire tal Enteresting, VEC
Date Extracted/Prepared: 8/27/85, 9/3/85	Separatory Furiel Extraction: YES
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO
ComplDit Footon A 11 G/FIAI	
Conc/Dil Factor: 0.11G/5ML	and the same of th

Number		ug.
319-84-6	Aiphe-BHC	<b>88</b> U
319-86-7	Bets-BHC	96 A
319-86-8	Delta-BHC	80 U
58-89-9	Gemme-BHC (Lindens)	<b>80</b> U
78-44-8	Heptschier	<b>80</b> U
308-00-2	Aldrin	50 U
1024-67-3	Heptachior Epoxide	<b>80</b> U
969-94-6	Endoculten i	100 U
60-67-1	Dieldrin	100 U
72-55-5	4,4'-DOE	100 U
72-29-8	Endrin	100 U
3321 3-65-8	Endoculturi II	190 U
72-54-8	4,4'-000	200 U
1031-07-0	Endocution Suifate	200 U
50-29-3	4,4'-DOT	200 U
72-43-5	Mother shlor	1000 U
53494-70-8	Friim Ketene	NA
57-74-0	Chlordene	1000 U
8001-35-2	Temphone	10000 U
12674-11-2	Arester-10100	NA
11104-28-2	Arector-1221	NA
11141-100-5	Aresier-1232	NA
63469-21-9	Arecler-1242	1000 U
12672-29-6	Aresier-1248	1000 U
11097-69-1	A:refer-1254	1000 U
11096-82-5	Arector-1260	1000 U

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub>= Volume of water extracted (ml)

Ws= Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

V<sub>S</sub> = NR or W<sub>S</sub> = 0.11

V. = 5000

1097

V. = 5

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7/85

CLF: 11/14/85

# Organics Analysis Data Sheet (Page 1)

Laboratory Na	me: <u>California Analytical La</u>	poratories, inc.		Case No: 214	113	
Lab Sample ID No: 21413-3		1	QC Report No: NR			
•	: SOIL			Contract No:		
•	Authorized By: P4	1		• • • • • • • • • • • • • • • • • • • •	Received: 6/21/85	
Data nelease	Authorized by.			Daie Sample	Tiecelvec. Mailes	
		Volatile C	Corr	pounds		
	Concentra	tion: Medlum				
	Date Extra	acted/Prepared:9/	16/8	5	-	
	Date Anal	yzed: <b>9/16/85</b>				
		actor: 100				
		loisture: NR				
	Percent M	loisture (Decanted	d): <b>N</b>	A		
CAS				CAS		
Number		ug/Kg		Number		ug/K
74 87-3	Chloromethene	200 U		78-87-6	1,2-Dichlaropropane	200 U
74-43.9	Bromomethane	200 U		10061-02-4	Trans-1,3-Dichloropropens	200 U
75.01-4	Vinyl Chlorida	200 U	Ĺ	79-01-4	Trichloroethene	200 U
- 00-3	Chloroethane	200 U		124-48-1	Dibromochiorom~thane	200 U
75-C9-2	Methylene Chloride	500 U		79-00-5	1,1,2-Trichioroethene	200 U
67-64-1	Acetona	500 U		71-43-2	Benzene	200 U
70-15- <b>0</b>	Carbon Disuifide	3C3 U	l	10061-01-5	cis-1,3-Dichloropropens	200 U
*) 35-4	1,1-Dichloroethene	200 U		110-75-8	2-Chioroethylvinylether	1000 U
75-34-3	1,1-Dichioroethane	200 U	Į	75-25-2	Bromoform	200 U
156-60-5	Trans-1,2-Dichioroethene	200 U	Į	100-10-1	4-Methyl-2-Pentanone	500 U
7-66-3	Chloroform	<b>200</b> U		591-78-6	2-Hexanone	500 U
107-06-2	1,2-Dichioroethane	200 U		127-18-4	Tetrachioroethene	200 U
/ 1 93- <b>3</b>	2-Butanone	500 U	į	79-34-8	1,1,2,2-Tetrachloroethane	200 U
1 35 6	1,1,1-Trichloroethmie	250	l	108-88-3	Toluene	200 U
23-5	Carbon Tetrac: loride	200 U		108-90-7	Chiorobenzene	200 U
.30-35-4	Vinyt Acetate	1000 U	ļ	100-41-4	Ethylbanzene	500 U
7 27 4	Bromodichioromethene	200 U	} t	100-42-5	Styrene	200 U
			Į		Total Xylenes	200 U
Data Reporting Qualifiers						
		results to EPA, the follow	wing r	esults qualifiers ar		
		is or footholes explainin schiftag must be explicit		wz ara auconieda	G. Flowerer, the	
Value	If the result is a value greater than or eq	ual to the		c	This flag applies to pestigde parameters to	
	detection firmit, report the value				identification has been confirmed by GC/N component pesticides >= 10ng/ul in the file	
ປ	Indicates compound was analyzed for bi Report the minimum detection limit for the				should be confirmed by GC/MS	
	the U (e.g. 100) based on necessary co- dilution actions. (This is not necessarily				This flag is used when the analyte is foundatively as well as a sample. It indicates possible	
detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The				blank contamination and warns the data u appropriate action.		
number is the minimum attainable detection limit for				THE PROPERTY OF THE PROPERTY O		
	the sample			Other	Other specific flags and footnotes may be	required to
J	Indicates an estimated value. This flag				properly define the results. If used, they ridescribed and such description attached to	nust be fully o the data
when estimating a connentration for tentatively identified compounds where a 1.1 response is assumed			NA	summary report. Not Analyzed		
or when the mass spectral data indicated the presence				# NR	See cover letter	570
	of a compound that meets the identificat the result is less than the specified delec-	tion limit but		NR S	Not Required. Spiked Compound.	•••
	greater than zero (e.g. 10J). If limit of de	Hection is 10ug/	940			

... 7 11/14/8**5** 

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repared by:

### Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

Concentration: LOW	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/10/85	Continuous Liquid - Liquid Extraction: NO
Conc/DiL Factor: 29G/ML	

#### CAS Numbe

CLF: 10/11/85

Number Number		ug/Kg
108-95-2	Phenol	200 U
111-44-4	bis(-2-Chloroethyl)Ether	200 U
95-57-8	2-Chlorophenoi	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlarobenzene	200 U
100-51-6	Benzyl Alcohol	200 U
95-50-1	1,2-Dichlorobenzene	200 U
95-48-7	2-Methylphenol	20C U
39638-32-9	bis(2-chlorolaspropyhEthor	400 U
106-44-5	4-Methylphonal	200 U
621-64-7	N-Nitrose-Di-n-Propylaminc	200 U
67-72-1	Hexachioroethene	200 U
96-95-3	Mirobenzene	206 U
78-59-1	leophorone	206 U
88-75-5	2-Nitrophenal	400 U
105-67-9	2,4-Dimethylphenal	206 U
65-65-0	Benzalc Acid	1000 U
111-81-1	bis(-2-Chioroethosy)Methene	400 U
120-83-2	2,4-Dichiarophenot	200 U
120-82-1	1,2,4-Trichlorobenzene	200 U
91-20-3	Naphthalene	200 U
106-47-8	4-Chlora niline	200 U
87-48-3	Hexachlorobuladiens	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
91-57-4	2-Methylnaphthalene	200 U
77-47-4	Hexachlorocyclopentadiene	200 U
88-06-2	2,4,6-Trichlarophenal	200 Ua
96-96-4	2,4,5-Trichlorophenal	
91-58-7	2-Chioronaphthalene	200 U
88-74-4	2-Nitroeniline	1000 U
131-11-3	Dimethyl Phthalate	200 U
206-96-8	Acenephthylene	200 U
99-09-2	3-Nitroeniline	1000 U

### CAS

Number		ug/Kg
83-32-6	Acensphthene	200 U
51-26-6	2,4-Dinitrophenol	1000 U
100-02-7	4-Nitrophenal	1000 U
132-44-9	Dibenzofuren	200 U
121-14-2	2,4-Ointrotoluene	408 U
606-20-2	2,6-Cinicroteluene	400 U
84-84-2	Dierhylpithelete	200 U
7005-72-3	4-Chlorophenyl-phenylether	200 U
84-73-7	Fluorane	200 U
100-01-6	4-Mitreanifine	1900 U
834-62-1	16-Dinkre-2-Mathylphenol	400 U
86-30-6	N-Nitroeodiphenylamine(1)	200 U
101-65-3	4-Bramophenyl-phenylether	200 U
118-74-1	Hexachiorobenzone	200 U
87-46-4	Pentachlorophenol	200 C
86-01-8	Phononthrone	200 U
120-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthelate	200 U
206-44-0	Fluorenthene	200 U
129-00-0	Pyrene	200 U
86-68-7	Buty:benzylphthelate	200 U
91-94-1	3,3'-Dichiorohenzidine	400 U
56-55-3	Senzo(a)Anthracene	200 U
117-81-7	bio(2-Ethylhexyl)Phthalate	200 U
218-01-8	Chrysene	400 U
117-84-0	Di-n-Octyl Phthelate	200 U
205-89-2	Benzo(b)Fluorenthene	400 U
207-08-9	Benzo(k)Fluorenthene	400 U
90-32-4	Senzo(a)Pyrene	400 U
193-39-5	Indena(1,2,3-cd)Pyrene	400 U
53-70-3	Dibenz(a,h)Anthracene	400 U
191-24-2	Senzo(g,h,i)Perylene	400 U

(1) - Cannot be separated from diphenylamine

Form I

poered by: 08

7/85

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# Organics Analysis Data Sheet (Page 3)

#### Pesticide/PCBs

Concentration: LOW	GPC Cleanup: NO
Data Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/18/85	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 1.54G/5ML	

Number		ug/
319-84-6	Alpha-BHC	1.0 U
319-85-7	Beta-BHC	<b>1</b> .0 U
219-86-8	Defta-BHC	3.0 U
58-89-9	Gamma-BHC (Lindana)	3.0 U
76-44-8	Heptanhlor	· 3.0 U
309-00-2	Aldrin	3.0 U
1024-87-3	Heptachlor Epoxide	1.0 U
059-96-6	Endosultan I	7.0 U
60-57-1	Dieldrin	7.0 U
72-55-8	4,4'-DDE	7.0 U
72-20-8	Endrin	7.0 U
33213-65-8	Endosultan II	7.0 U
72-54-8	4,4'-000	13 U
1031-07-8	Endosultan Sulfate	13 U
50-29-3	4,4'-DDT	13 U
72-43-5	Methoxychior	67 U
53494-70-5	Endrin Ketone	NA
57-74-0	Chlordane	67 U
1001-35-2	Toxaphene	670 U
12674-11-2	Arocior-1016	NA
11104-28-2	Aroclor-1221	NA
11141-16-5	Aroclor-1232	NA NA
53469-21-9	Aroclor-1242	67 U
12672-29-6	Arocior-1248	67 U
11097-69-1	Arocior-1254	67 U
11096-82-5	Aroclor-1260	87 U

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub> = Volume of water extracted (ml)

W<sub>s</sub> = Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

V<sub>s</sub> = NR

or W<sub>S</sub> = 1.54

654

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7/85

CLF 11/14/85

Prepared by:

### **Organics Analysis Data Sheet** (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.	Case No: 21591	
Lab Sample ID No: 21591-5	QC Report No: NR	
Sample Matrix: SQIL	Contract No: MR	
Data Release Authorized By:	Date Sample Received: 6/29/85	
Volatile C	ompounds	
Concentration: Maditum		
Date Extracted/Prepared: 9/	16/85	
Date Analyzed: 9/16/85		
Conc/Dil Factor: 100	H:NR	
Percent Moisture: NR		
Percent Moisture (Decanted	i): NR	

CAS

Number

BURNESS BURNESS

Mulliper		UQ/Kg
74-87-3	Chloremethene	200 U
74-83-9	Bromemethene	200 U
78-01-4	Vinyl Chloride	200 U
75-00-3	Chiercethene	200 U
75-00-2	Mathylene Chloride	800 U
67-64-1	Acetone	900 U
79-15-0	Carbon Disuffide	200 U
75-35-4	1,1-Dichloraethene	200 U
75-34-3	1,1-Dishlereethane	200 U
156-60-6	Trans-1,2-Dichloresthene	200 U
67-44-3	Chleroform	200 U
107-08-2	1,2-Dichle: sethene	200 U
78-83-3	2-Butanene	900 U
71-58-4	1,1,1-Trichloroethane	200 U
86-23-5	Carbon Tetrsubloride	200 U
108-06-4	Vinyi Acetate	1000 U
75-27-4	Bromedichloromethene	206 U

CAS

Number		ug/l
78-87-8	1,2-Dichleropropene	200 U
10061-02-6	Trans-1,3-Dichloreprepens	200 U
79-01-6	Trichlereathene	200 U
124-46-1	Disremechloromethene	200 U
79-00-5	1,1,2-Trichloreethene	200 U
71-43-2	Bortzorro	200 U
10061-01-8	ele-1,3-Dichlereprepene	200 U
110-75-8	3-Chioreethylvinylether	1000 U
78-26-2	Bremeterm	200 U
108-10-1	4-Mathy4-2-Pentanene	800 U
801-79-6	2-Hexanone	900 U
127-18-4	Tetrachieraethene	200 U
79-34-3	1,1,2,2-Tetrschieroethene	200 U
100-00-3	Teluene	200 U
108-60-7	Chlerebonzene	200 U
100-41-4	Ethylbenzene	900 U
100-42-5	Styrene	200 U
	Tetal Xylenes	200 U

### **Date Reporting Qualiflors**

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

Value If the result is a value greater than or equal to the detection limit, report the value.

- Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g., 10U) based on necessary concentration/ diution actions. (This is not necessarily the instrument detection limit.) This tootnote should read: U.-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g. 10J), if limit of detection is 10ug/l and a concentration of Sug/l is calculated, report as 3J.

- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >== 10 ng/// in the final extract should be confirmed by GC/MS.
- This flag is used when the analyse is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.

Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data

Summery report,
Not Analyzed,
See cover letter,
Not Required,
Spiked Compound,

265

Form I

Prepared by:

10/85

CLF: 11/14/85

# Organics Analysis Data Sheet (Page 2)

#### Semivolatile Compounds

Concentration: Low	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/12/85	Continuous Liquid - Liquid Extraction: NO
ConstDit Foster: 29G/MI	· · ·

CAS		
Number		ug/Kg
108-95-2	Phenol	200 U
111444	bis(-2-Chloroethyl)Ether	200 U
94-57-8	2-Chlorophenol	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlorobenzene	200 U
100-51-8	Benzyl Alcohol	200 U
95-50-1	1,2-Dichlorobenzene	200 U
35-48-7	2-Methylphenol	200 U
39638-32-0	bis(2-chloroisopropyi)Ether	400 U
106-44-5	4-Methylphenol	200 U
821-64-7	N-Nitroso-Di-n-Propylamine	200 U
67-72-1	Hexac. 'proethane	200 U
98-95-3	Nitrobenzene	200 U
78-52-1	Isophorone	200 U
88-75-5	2-Nitrophenol	400 U
105-67	2,4-Dimethylphenol	200 U
55-A5-O	Benzoic Acid	1000 U
111-01-1	bis(-2-Chioroethoxv)Methane	4C0 U
12(-83-2	2,4-Dichlorophenol	2 X0 U
120-82-1	1,2,4-Trichlorobenzene	3430
91-20-3	Naphthelene	20U U
105 47-8	4-Chloroeniline	200 U
97 5 <b>8-3</b>	Hexachiorobutadiena	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
7-6	2-Methylnephthelene	200 U
71474	Hexachlorocyclopentedlena	200 U
38-06-2	2,4,5-Trichlorophenol	200 U #
95-95-4	2,4,5-Trichlorophenol	•
91 58-7	2-Chioronaphthelene	200 U
23-74-4	2-Ntroaniline	1000 U
131-11-3	Dimethyl Phthalate	200 U
208-96-8	Acenephthylene	200 U
33-09-2	3-Nitroaniline	1000 U

CAS Number		ug/Kg
83-32-9	Acenaphthene	200 U
51-28-5		
	2,4-Dinitrophenol	1000 U
100-02-7	4-Nitrophenol	1000 U
132-64-9	Dibenzoturen	200 U
121-14-2	2,4-Dinitrotoluene	400 U
606-20-2	2,6-Dinitrotoluene	400 U
94-68-2	Diethylphthalate	200 U
7005-72-3	4-Chlorophenyl-phenylether	200 U
86-73-7	Fluorene	200 U
100-01-6	4-Nitroeniline	1000 U
534-52-1	4,6-Dinitro-2-Methylphenol	400 U
86-30-6	N-Nitrosodiphenylamins(1)	200 U
101-55-3	4-Bromop :enyl-phenylether	200 U
118-74-1	Hexachiorobenzene	200 U
87-86-6	Pentachlorophenol	200 U
85-01-8	Phenanthrene	200 U
120-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthalate	200 U
206-14-0	Fluorenthene	200 U
129-00-0	Pyrene	200 U
85-68-7	Butylbenzylphthelate	200 U
91-94-1	3,3'-Dichiprobenzidine	400 U
56-55-3	Benzo(a)Anthracene	200 U
117-81-7	bls(2-Ethylhexyl)Phthalate	200 U
218-01-9	Chrysene	400 U
117-94-0	Di-n-Octyl Phthalate	200 U
205-99-2	Benzo(b)Fluorenthene	400 U
207-08-9	Benzo(k)Fluoranthene	400 U
50-32-8	Benzo(a)Pyrene	400 U

(1) - Cannot be separated from diphenylamine

CLF 10/11/85

PROGRAM STANDARD OF STANDARD S

Form I Prepared by: \_

193-39-5

53-70-3

22\_\_\_

7/85

400 U

400 U

### Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

7 000000000	
Concentration: LOW	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO
Conc/Dil Factor: 1.5G/5ML	

Number		ug/K(
319-84-6	Alpha-BHC	3.0 U
319-86-7	Beta-BHC	2.0 U
319-00-0	Delta-BHC	1.0 U
50-09-0	Gemme-BHC (Lindane)	3.0 U
78-44-8	Heptachier	1.0 U
308-00-2	Aldrin	3.0 U
1024-67-3	Hectschier Epoxide	3.0 U
100-06-0	Endocutton I	7.0 U
66-67-1	Dieldrin	7.9 U
72-45-0	4,4'-00E	7.0 U
72-20-8	Endrin	7.0 U
33213-66-0	Endosulfen II	7.0 U
72-64-8	4,4'-000	13.0 U
1031-07-8	Endesulten Sulfate	12.0 U
80-25-3	4,4'-DOT	13.0 U
72-45-6	Metherychier	67,0 U
83494-70-8	Endrin Ketene	NA
57-74-0	Chierdane	67,0 U
8001-38-2	Temphone	670 U
12674-11-2	Arector-1016	NA
11104-28-2	Arecler-1221	NA
11141-16-6	Arestor-1232	NA
53469-21-0	Arector-1242	67.0 U
12672-29-6	Aresier-1248	67.0 U
11007-60-1	Arecter-1254	67.0 U
11096-82-6	Arector-1250	67.0 U

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub>= Volume of water extracted (ml)

Wg= Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

V<sub>s</sub> = NR

or W<sub>s</sub> =1.5

V<sub>t</sub> = 5000

V:=5 1135

267

Form

7/85

CLF: 11/14/85

Prepared by:

### Organics Analysis Esta Sheet (Page 1)

Laboratory Name: California Analytical I	aboratories, Inc. Case No: 21591
Lab Sample ID No: 21591-6	QC Report No: MR
Sample Matrix: SOIL	Contract No: NFt
Data Release Authorized By:	Date Sample Received: 5/29/85
	Volatile Compounds
Concer	tration: Medium
Date Ex	stracted/Prepared: 9/16/85
Date Ar	nalyzed: 9/16/85
Conc/D	il Factor: 100 pH:NR
Percent	Moisture: NR
Percent	Moisture (Decanted): NR
CAS	CAS

CAS		
Number		ug/K
74-87-3	Chloromethane	200 U
74-33	Bremomethene	200 U
75-01-4	Vinyt Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	500 U
67-64-1	Acetone	800 U
75-15-0	Carbon Disulfide	200 U
73-35-4	1,1-Dichloroethene	200 U
75-34-3	1,1-Dichioroethane	200 U
156-60-5	Trans-1,2-Dichlorosthene	200 U
17-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-93-3	2-Butanone	500 U
71-55-6	1,1,1-Trichioroether e	200 U
**-23-5	Carbon Tetrachroride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		
Manual		ug/K
78-87-6	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichioropropens	200 U
79-01-6	Trichioroethene	200 U
124-46-1	Dibromochloromethane	290 U
79-06-5	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-5	cis-1,3-Dichloropropens	200 U
110-75-6	2-Chloroethylvinylether	1000 U
75-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	800 U
591-78-6	2-Hexanone	500 U
127-18-4	Tetrachloroethene	200 U
79-34-5	1,1,2,2-Tetrachioroethane	200 U
106-68-3	Toluene	200 U
108-80-7	Chloroberizene	200 U
100-41-4	Ethylbenzene	500 U
100-42-6	Styrene	200 U
	Total Xvienee	200 LI

#### **Data Reporting Qualifiers**

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of cach flag must be explicit.

Value If the result is a value greater than or equal to the detection limit, report the value.

Indicates compound was analyzed for but not detected Report the minimum detection limit for the sample with the U is g. 10U) based on necessary concentration, diubon actions. (This is not necessarily the instrument detection limit.) The footnote should read, U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.

Indicates an estimated value. This flag is used either when estimating a concentration for tenhativery identified compounds where a 1.1 response is assumed or which the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10J). If I mit of detection is 10ug/I and a concentration of Sug/I is calculated, report as 3J.

- This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >== 10ng/ul in the final extract should be confirmed by GC/MS.
- This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warms the data user to take

Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data

See cover letter.
Not Required.
Spiked Compound. NR

1167

1. F 11/14/85

Form I

10/85

268





### Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

Concentration: Low	GPC Cleanup: NO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/12/85	Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	200 U
111-44-4	bis(-2-Chloroethyl)Ether	200 U
96-57-9	2-Chlorophenol	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlerobenzene	300 U
100-51-6	Benzyl Alcohol	200 U
96-50-1	1,2-Dichloroberizene	200 U
96-48-7	2-Methylphenol	200 U
39636-32-0	bis(2-chloroleopropyl)Ether	400 U
106-44-8	4-Methylphenol	200 U
621-64-7	N-Mitroeo-Di-n-Propylemine	200 U
67-72-1	Hezechleroethene	200 U
90-05-3	Nitrobenzone	200 U
79-59-1	Isophorone	200 U
88-75-5	2-Nitrophenol	400 U
105-67-9	2,4-Dimethylphenol	200 U
65-85-0	Benzolc Acid	1006 U
111-91-1	bie(-2-Chloroethoxy)Methene	400 U
120-83-2	2,4-Dichlarophenal	200 U
120-82-1	1,2,4-Trichlorobenzene	200 U
91-20-3	Naphthelene	200 U
106-47-8	4-Chloroeniline	200 U
87-68-3	Hexachiorobutadiene	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
91-57-6	2-Methylnaphthalene	200 U
77-47-4	Hexachioracyclopentadiene	200 U
88-06-2	2,4,6-Trichiorophenal	200 U #
95-95-4	2,4,5-Trichlorophenal	•
91-58-7	2-Chloronephthalene	200 U
88-74-4	2-Nitroeniilne	1000 U
131-11-3	Dimethyl Phthalate	200 U
208-96-8	Acenaphthylene	200 U
99-09-2	3-Nitroeniline	1000 U

Conc/Dil Factor: 29G/ML

CAS Number		ug/Kg
83-32-0	Acenephthene	200 U
81-28-8	2,4-Dinitrophenol	1000 U
100-02-7	4-Mitrophenol	1000 U
132-44-9	Other: tofuren	200 U
121-14-2	2,4-Dinitrotoluene	400 U
606-20-2	2,6-Dinitrotoluene	400 U
84-86-2	Disthylphthalate	ن ويو
7005-72-3	4-Chlorophenyl-phenylether	200 U
84-73-7	Rugrens	200 U
100-01-6	4-Nitroeniline	1000 U
834-62-1	4,6-Dinitre-2-Methylphenol	400 U
86-30-4	N-Nitroecdlphenylemine(1)	200 U
101-66-3	4-Bromophenyl-phenylether	200 บ
118-74-1	Hexachlorobenzene	200 U
87-86-6	Pentachiorophenol	200 U
85-01-8	Phenanthrene	200 U
120-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthelate	200 U
206-44-0	Fluorenthene	200 U
129-00-0	Pyrone	200 U
86-68-7	Butyfbenzylphthelate	200 U
91-94-1	3,3'-Dichlorobenzidine	400 U
96-66-3	Benzo(a)Anthracene	200 U
117-81-7	ble(2-Ethylhexyl)Phthelate	200 U
218-01-0	Chrysene	400 U
117-84-0	Di-n-Octyl Phthelate	200 U
205-88-2	Benzo(b)Fluoranthene	400 U
207-08-9	Benzo(k)Fluoranthene	400 U
50-32-8	Benzo(a)Pyrene	400 U
193-39-5	Indeno(1,2,3-cd)Pyrene	400 U
53-70-3	Dibenz(a,h)Anthracene	460 U
191-24-2	Benzo(g,h,i)Perylene	400 U

Connot be separated from diphenylamine

1168

CLF: 10/11/85

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### **Organics Analysis Data Sheet** (Page 3)

#### Pasticide/PCBs

Concentration: LOW	GPC Cleanup: INO
Date Extracted/Prepared: 8/27/85	Separatory Funnel Extraction: YES
Date Analyzed: 9/19/85	Continuous Liquid - Liquid Extraction: NO

CAS

Number 319-84-4 Alpha-8HC 3.0 U 319-85-7 Bets-8HC 3.0 U 319-86-8 Deltu-BHC 3.0 U 58-89-9 Gamma-BHC (Lindane) 3.0 U 76-44-8 Heptschlor 3.0 U 309-00-2 Aldrin 3.0 U 1024-57-3 3.0 U Heptachlor Epoxide 969-96-8 7.0 U Endosultan I 60-57-1 Dieldrin 7.0 U 72-55-0 4,4'-DOE 7.0 U 72 20-8 Endrin 7.0 U 33213-65-9 Endosultan II 7.0 U 72-54-8 4.4'-000 13.0 U 1031-07-8 Endosulfan Sulfate 13.0 U 50-29-3 4,4'-DOT 13.0 U 72-43-5 Methoxychlor 67.0 U 53494-77-5 NA Endrin Katone 57-74-0 Chlordene 67.0 U 8001-35-2 Toxaphene 670 U 12674-11-2 Arocior-1016 NA 11104-28-2 Aroclor-1221 NA 11141-16-5 Aroclor-1232 NA 53469-21-9 Aroclor-1242 67.0 U 12672-29-6 Arocior-1248 67.0 U 11097-69-1 Aroclor-1254 67.0 U 11096-82-5 Arocior-1260 67.0 U

V<sub>i</sub> = Volume of extract injected (ul)

V<sub>s</sub> = Volume of water extracted (ml)

Ws= Weight of sample extracted (g)

V<sub>t</sub> = Volume of total extract (ul)

 $V_s = NR$  or  $W_s = 1.5$ 

V<sub>t</sub> = 5000

 $v_{i=5}$  1169

CLF. 11/14/85

Conc/Dil Factor: 1.5G/5ML

HU-NCBC-RI-OI

### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C25-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

COW	NAME: CALIF.AN NO.: 784		CASE NO: 2159/ QC RPT. 2/54/	-
LAB	SAMPLE NO.:	21591-1	DATE: 3-14-16	
	ELEMENTS	: IDENTIFIED	AND MEASURED	
	•			

MATRIX: SOIL	UNITS	: MG/KG DRY WEIGHT
ELEMENTSMETHOD  2. ANTIMONYP  3. ARSENICP	<u> &lt;3y</u> R	
5. BERYLLIUMP 6. CADMIUMP 8. CHROMIUMP	<0.30 <0.30 7.3	

10.COPPER....P 12.LEAD......P 15.MERCURY....CV 16.NICKEL.....P

18.SELENIUM....P 19.SILVER....P 21.THALLIUM....F

24.ZINC......P 25.CYANIDE....C

#### COMMENTS:

ICP Interelement and background corrections applied?

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

#### FOOTNOTES:

NR - not required by contract at this time
Value - If the result is a value greater than or equal to the
instrument detection limit but less than the contract required detection limit, report the value in brackets (ie.[10]).

Indicate the analytical method used with P (for ICP/Flame AA),

F (for furnace), or CV (for cold vapor).

U - Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <100).

- Indicates a value estimated or not reported dua to the presence of

interference. Explanatory note included on cover page. S - Indicates value determined by Method of Standard Addition.

R - Indicates spike sample recovery is not within control limits.
 \* - Indicates duplicate analysis is not within control limits.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995.

HU-NCBC-R2-01

#### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

LAB NAME: CALIF.ANAL.LAB. CASE NO: QC RPT.# SOW NO.: 784 LAB SAMPLE NO.: DATE:

> ELEMENTS IDENTIFIED AND MEASURED

Ja12 UNITS: MG/KG MATRIX: DRY WEIGHT

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#### COMMENTS:

ICP Interelement and background corrections applied?

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections applied before generation of raw data.

#### FOOTNOTES:

MR - not required by contract at this time Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie.[10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).

- Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <10U).

- Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page. - Indicates value determined by Method of Standard Addition.

Indicates spike sample recovery is not within control limits.
Indicates duplicate analysis is not within control limits.
Indicates the correlation coefficient for method of standard addition is less than 0.995.

FORM V

## SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

DATE:	3-19-86	HU-NO	BC-R2-01
MATRIX:	YOIL	UNITS:	ppb
	0011mm 4 =		

COMPOUNDS METALS: ELEMENTSMETHO	CONTROL LIMIT * R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)
2. ANTIMONY. P 3. ARSENIC. P 5. BERYLLIUM. P 6. CADMIUM. P 8. CHROMIUM. P 10.COPPER. P 12.LEAD. P 15.MERCURY. CV 16.NICKEL. P 18.SELENIUM. P 19.SILVER. P 21.THALLIUM. F 24.ZINC. P	75 TO 125 75 TO 125	53.3 408 92.3 84.6 45.1 35.8 44.8 NIR 154 7.1 61 22.8 8760 878	24.4 /09 <20 <20 /22 /23 ,300 N/R 31.7 <20 <20 <20 <20 /20 /20 /20 /20 /20 /20 /20 /	200 360 100 100 200 200 100 100 200

COMMENTS:

#### FM VI

#### DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

HU-NOBC-R2-01

DATE: MATRIX:

UNITS: ppb

COMPOUNDS METALS:

CONTROL SAMPLE(S) DUPLICATES(D) LIMIT

RPD

ELEMENTS..METHOD 2. ANTIMONY....P

3. ARSENIC....P 5. BERYLLIUM...P 6. CADMIUM....P

8. CHROMIUM....P 10.COPPER....P

12.LEAD....P 15.MERCURY...CV

16.NICKEL.....P

18.SELENIUM....P 19.SILVER.....P

21. THALLIUM....F 24.ZINC.....P 25.CYANIDE....C

COMMENTS:

HU-NCBC-RI-02

#### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

LAB NAME: CALIF.ANAL.LAB.

SOW NO.: 784

LAB SAMPLE NO.: 2/4/3-2

DATE: 2-19-2/2

ELEMENTS IDENTIFIED AND MEASURED

16.NICKEL....P

18.SELENIUM...P

19.SILVER....P

21.THALLIUM...F

24.ZINC...P

24.ZINC....P  $\begin{array}{ccc}
27 \\
\hline
25.CYANIDE....C
\end{array}$ 

#### COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

#### FOOTNOTES:

NR - not required by contract at this time

Value - If the result is a value greater than or equal to the

instrument detection limit but less than the contract required

detection limit, report the value in brackets (ie.[10]).

Indicate the analytical method used with P (for ICP/Flame AA),

F (for furnace), or CV (for cold vapor).
U - Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <100).

E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

interference. Explanatory note included on cover page.

S - Indicates value determined by Method of Standard Addition.

R - Indicates spike sample recovery is not within control limits. \* - Indicates duplicate analysis is not within control limits.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995.

## SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

3-19-86	HU-NCBC-R1-02	
	UNITS: ppb	
		3-77-86

COMPOUNDS METALS: ELEMENTSMETHOR	CONTROL LIMIT * R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	
2. ANTIMONY P 3. ARSENIC P 5. BERYLLIUM P 6. CADMIUM P 8. CHROMIUM P 10.COPPER P 12.LEAD P 15.MERCURY CV 16.NICKEL P 18.SELENIUM P 19.SILVER P 21.THALLIUM F 24.ZINC P	75 TO 125 75 TO 125	39   30,4 00.6 00.7 211 628 N/R 264 11.5 15.8 30.7 5770 N/R	29 173 420 120 124 59.0 183 20 420 420 420 420 420 420 420	200 300 100 400 200 200 200 200 200 200 200 200	

OMMENTS:

#### FM VI

#### DUPLICATE SAMPLE RECOVERY

EG & G ID NO.: LAB NAME: CALIF. ANAL. LABS. HU-NEBC-RI-02 DATE: UNITS: ppb COMPOUNDS CONTROL SAMPLE(S) DUPLICATES(D) RPD METALS: LIMIT ELEMENTS..METHOD 2. ANTIMONY....P 3. ARSENIC....P 5. BERYLLIUM...P 6. CADMIUM....P 8. CHROMIUM....P 10.COPPER.....P 12.LEAD.....P 15.MERCURY....CV 16.NICKEL....P 18.SELENIUM...P 19.SILVER....P 21.THALLIUM...F 24.ZINC.....P

COMMENTS: Antimony and nickel were not flagged with an "\*" for high RPD because the values were only at the detection limits.

25.CYANIDE....C

HU-NCBC-R2-02

#### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

21591 LAB NAME: CALIF.ANAL.LAB. CASE NO: QC RPT. SOW NU.: 784 LAB SAMPLE NO.: DATE: ELEMENTS IDENTIFIED AND MEASURED UNITS: MG/KG DRY WEIGHT ELEMENTS. METHOD 2. ANTIMONY....P 3. ARSENIC....P 5. BERYLLIUM...P 6. CADMIUM....P 8. CHROMIUM....P 10.COPPER.....P 12.LEAD.....P 15.MERCURY....CV 16.NICKEL....P 18.SELENIUM....P 19.SILVER.....P 21.THALLIUM....F 24.ZINC.....P 25.CYANIDE....C

#### COMMENTS:

Interelement and background corrections applied? Yas.

porrections consist of Zeeman effect background correction Perkin-Elmer 3030 instruments and correction of background apption by D2 lamp on Varian 875 instruments. Corrections applied before generation of raw data.

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not required by contract at this time

If the result is a value greater than or equal to the instrument detection limit but less than the contract/required detection limit, report the value in brackets (ie.[10]).

Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).

Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).

Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

Indicates value determined by Method of Standard Addition.

Indicates spike sample recovery is not within control limits.

Indicates duplicate analysis is not within control limits.

Indicates the correlation coefficient for method of standard addition is less than 0.995.

9. 2004 9.

FORM V

#### SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. AWAL. LABS.	• •	EG	& G ID	
DATE: 3-19-86 MATRIX: <u>3(14</u>	•		- <i>NIBC - R2 - C2</i> ITS: ppb	2
COMPOUNDS CONTROL LIMIT R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	
ELEMENTSMETHOD  2. ANTIMONYP 75 TO 125  3. ARSENICP 75 TO 125  5. BERYLLIUMP 75 TO 125  6. CADMIUMP 75 TO 125  8. CHROMIUMP 75 TO 125  10.COPPERP 75 TO 125  12.LEADP 75 TO 125  15.MERCURYCV 75 TO 125  16.NICKELP 75 TO 125  18.SELENIUMP 75 TO 125  19.SILVERP 75 TO 125  21.THALLIUMF 75 TO 125  24.ZINCP 75 TO 125  25.CYANIDEC 75 TO 125	243 70 9 73 4 4108 320 272 218 247 216 3 216 3	400 -27 -20 -20 -20 -20 -20 -20 -20 -20	200 300 100 100 400 250 50 50 50 500	2 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5

:OMMENTS:

#### DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID No.:

DATE: 3-/9-8%
MATRIX: 50:4

HU-NEBC-RI-CI

UNITS: ppb

COMPOUNDS METALS:

STATES STATES AND STATES OF SECONDARY STATES AND STATES

CONTROL SAMPLE(S) DUPLICATES(D) LIMIT

RPD

ELEMENTS.METHOD

2. ANTIMONY...P

3. ARSENIC...P

5. BERYLLIUM...P

6. CADMIUM...P

8. CHRONIUM...P

6. CADMIUM. P
8. CHRONIUM. P
10.COPPER. P
12.LEAD. P
15.MERCURY. CV
16.NICKEL. P
18.SELENIUM. P
19.SILVER. P
21.THALLIUM. F
24.ZINC. P

25.CYANIDE....C

<u> 32.9</u>	<20
	27
<u> </u>	<20
<del>- 520</del>	<u> &lt;20</u>
1103	1100
<u></u>	100
	1.51
NI'R	$\omega/\phi$
	57.4
<u> </u>	<u> </u>
	<u> &lt;30</u>
- Sai ()	
<u> </u>	1200
	$\mathcal{L}^{i}\mathcal{R}^{i}$

.OMMENTS:

HU-NCBC-R2-03

# MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

LAB NAME: CALIF.ANAL.LAB. CASE NO: SOW NO .: OC RPT. 784 21501-4 LAB SAMPLE NO.:

> ELEMENTS IDENTIFIED AND MEASURED

5012 MG/KG DRY WEIGHT

ELEMENTSMETHOD	
2. ANTIMONYP	<30 R
3. ARSENICP	1,3
5. BERYLLIUMP	<(), (2.1)
6. CADMIUMP	2,8
8. CHROMIUMP	2/
10.COPPERP	29
12.LEADP	<u> </u>
15.MERCURYCV	<u> </u>
16.NICKELP	- 49
19.SILVERP	<u> &lt;11,311</u>
21.THALLIUMF	-221
24.ZINCP	<u> </u>
25.CYANIDEC	40.511

#### COMMENTS:

ICP Interelement and background corrections applied?

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

#### FOOTNOTES:

NR - not required by contract at this time Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie.[10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).

U - Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <100).
Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page. Indicates value determined by Method of Standard Addition.

R - Indicates spike sample recovery is not within control limits.
 \* - Indicates duplicate analysis is not within control limits.

Indicates the correlation coefficient for method of standard addition is less than 0.995.

HU-NCBC-RY-09

## MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

21413 LAB NAME: CALIF.ANAL.LAB. CASE NO: 21413 QC RPT.# SOW NO.: 784 LAB SAMPLE NO.: 21413-3 DATE: 2-14-86

> ELEMENTS IDENTIFIED AND MEASURED

MARCOAL MG/KG UNITS: MATRIX: DRY WEIGHT

ELEMENTSMETHOD 2. ANTIMONYP	<3.1 R
3. ARSENICP	20.51)
5. BERYLLIUMP	<u> </u>
6. CADMIUMP	<u> </u>
10.COPPERP	<1,21)
12.LEADP	7.5
15.MERCURYCV 16.NICKELP	$\frac{\langle 0,11\rangle}{\langle 0,11\rangle}$
18.SELENIUMP	- 20.30 P
19.SILVERP	<0.50 R
21.THALLIUMF	<u> &lt;1,5U</u> R
24.ZINCP 25.CYANIDEC	<del>- 19                                   </del>

#### COMMENTS:

109 Interelement and background corrections applied?

AA corrections consist of Zeeman effect background correction 20 Parkin-Elmer 3030 instruments and correction of background approprian by D2 lamp on Varian 875 instruments. Corrections expplied before generation of raw data.

#### TO ANTEDTES:

not required by contract at this time for the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie.[10]).

Indicate the analytical method used with P (for ICP/Flame AA),
F (for furnace), or CV (for cold vapor).

Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <10U).

Indicates a value estimated or not reported due to the presence of

interference. Explanatory note included on cover page. Undicates value determined by Method of Standard Addition. Indicates spike sample recovery is not within control limits. Indicates duplicate analysis is not within control limits.

Indicates the correlation coefficient for method of standard addition is less than 0.995.

1995

FORM V

#### SPIKE SAMPLE RECOVERY

eg & G ID

HU-NCBC	-R1-09	
,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	• • • • • •	

DATE:	3-19-86
MATRIX:	MARINAL

UNITS: ppb

COMPOUNLS METALS:	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	* R
ELEMENTSMETHO		/. A	220	2.40	<b>~</b>
2. ANTIMONYP	75 TO 125	60	<u> </u>	200	_30
3. ARSENICP	75 TO 125	40.5	<del>- 430</del> <del>- 430</del>	<del>- 50</del>	<del></del>
5. BERYLLIUMP	75 TO 125 75 TO 125	97.7	<del>- \$20</del>	/6D	<u> </u>
8. CHROMIUMP	75 TO 125	218	37.7	200	<del>- 7/2</del>
10.COPPERP	75 TO 125	43.2	₹20	100	93
12.LEADP	75 TO 125	1070	165	1000	91
15.MERCURYCV	75 TO 125	NIR	NIR		
16.NICKELP	75 TO 125	125	43.4		4
18.SELENIUMP	75 TO 125	15,2	<del>_&lt;20</del>		-30
19.SILVERP	75 TO 125	- S 2 · 4	<u> </u>	100	_25
21.THAJLIUMF	75 TO 125	74.4	374	<u> 50</u>	<del></del>
25.CYANIDEC	75 TO 125	N.R	NIR		
				_	-

COMMENTS:

#### DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID No.:

HU-NCBC-RI-09

UNITS: ppb

COMPOUNDS METALS:

CONTROL SAMPLE(S) DUPLICATES(D) LIMIT

RPD

LEMENTS. METHOD
ANTIMONYP
ARSENICP
BERYLLIUMP
6. CADMIUMP
" CHROMIUM. P
COPPERP
L2.LEAD. P
5 MERCURYCV
I MERCURICV
15. NICKELP
18.SELENIUMP
19.SILVERP
21.THALLIUMF

ANIDE ....C

<u> </u>	< <u>১</u> ০ _<১০
<u> </u>	<20
<20	<20
<20	< 3.0
<u> </u>	<u> </u>
1/0.5	(2/2
NIR	NIR.
43.4	45
- <del>\leq 2</del> \leq - \leq 2 \leq -	<20
<del>- 230</del>	<u> </u>
374	307
NIR	W/R

DEFENTS:

HU-NCBC-R2-09

#### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

LAB NAME: CALIF.ANAL.LAB. CASE NO: QC RPT.# 78; SOW NO.: 21591-5 DATE: LAB SAMPLE NO.:\_

> ELEMENTS IDENTIFIED AND MEASURED

MARCORL UNITS: MG/KG MATRIX: DRY WEIGHT

_
<3U R
[1.0]
<0.3 U
< 0.3 ()
4.9
1.2
8,3
20.10
2.4
<0.30 K
<0.5U R
40,30 R
16
<0.5 U

#### COMMENTS:

ICP Interelement and background corrections applied?

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

#### FOOTNOTES:

NR - not required by contract at this time

Value - If the result is a value greater than or equal to the

instrument detection limit but less than the contract required

detection limit, report the value in brackets (ie.[10]).

Indicate the analytical method used with P (for ICP/Flame AA),

F (for furnace), or CV (for cold vapor).

U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).

- Indicates a value estimated or not reported due to the presence of
- interference. Explanatory note included on cover page.
   Indicates value determined by Method of Standard Addition.
- R Indicates spike sample recovery is not within control limits.
   \* Indicates duplicate analysis is not within control limits.
   + Indicates the correlation coefficient for method of standard addition is less than 0.995.

## SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

DATE: 3-19-86
MATRIX: 14-0011CA1

HU-NC3C- R2-09

UNITS: ppb

COMPOUNDS  (FTALS:	CONTROL LIMIT * R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)
ANTIMONY. P AMSENIC. P E. EURYLLIUM. P 6. CADMIUM. P CHROMIUM. P 10. COPPER. P 12. LEAD. P 15. MERCURY. CV 10. MICKEL. P 6 SELENIUM. P 19. SILVER. P	75 TO 125 75 TO 125	22.2 45.4 47.9 90.3 426 106 608 N/R 280 16.2 51.5 27.9 22.9 N/R	22.7 20.8 200 230 27.0 24.2 106 20 20 20 20 20 213 218	260 400 400 400 700 500 500 700 500

WENTS:

#### DUPLICATE SAMPLE RECOVERY

AB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

	HU-NCBC-R2-09
1	

DATE: 3-19-86 MATRIX: CHORCOAL

UNITS: ppb

COMPOUNDS METALS:	CONTROL SAMPLE(S) LIMIT	DUPLICATES (D)	RPD
ELEMENTS. METHOD  2. ANTIMONY. P  3. ARSENIC. F  5. BERYLLIUM. P  6. CADMIUM. P  8. CHROMIUM. P  10. COPPER. P  12. LEAD. P  15. MERCURY. CV  16. NICKEL. P  18. SELENIUM. P  19. SILVER. P  21. THALLIUM. F  24. ZINC. P	22 7 20.8 20.8 220 21.0 21.6 1	<20 22.6 <20 <20 /01 .65,2 169 .07 .07 .07 .07 .07 .07 .07 .07	C

OMMENTS:

HU-NOBO-RO-OGA

#### MODIFIED PRIORITY POLLUTANT LIST (EG & G Subcontract No. C85-130761-KAM-177-85) INORGANIC ANALYSIS SHEET

CASE NO: LAB NAME: CALIF.ANAL.LAB. QC RPT. 784 SOW NO.: 2-10-41 DATE: LAB SAMPLE NO.:

ELEMENTS IDENTIFIED AND MEASURED

(LARMA! UNITS: MG/KG MATRIX: DRY WEIGHT

ELEMENTSMETHOD	
2. ANTIMONYP	_ < 3U R
3. ARSENICP	2.5
5. BERYLLIUMP	K/1 311
6. CADMIUMP	< 2,30
8. CHROMIUMP	3
10.COPPERP	1.5
12.LEADP	<i>i</i> • •
15.MERCURYCV	<:.nU
16.NICKELP	[1, x]
18.SELENIUMP	< 0.3UR
19.SILVERP	<0.50 R
21.THALLIUMF	<0.50 R
24.ZINCP	14
25.CYANIDEC	<i>∠0.50</i>

#### COMMENTS:

TOP Interelement and background corrections applied?

As corrections consist of Zeeman effect background correction A Perkin-Elmer 3030 instruments and correction of background corption by D2 lamp on Varian 375 instruments. Corrections applied before generation of raw data.

#### THOTES:

not required by contract at this time

wille - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie.[10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
Indicates element was analyzed for but not detected. Report with

the detection limit value (e.g., <10U). Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

Endicates value determined by Method of Standard Addition.

Indicates spike sample recovery is not within control limits. Indicates duplicate analysis is not within control limits. - Indicates the correlation coefficient for method of standard

addition is less than 0.995.

2009

## 2,4-D and 2,4,5-T by EPA Method 8150

-1	CAL I.D. 21349-MB	mg/Kg (ppm) 2,4-D <0.01	BE/KE (PPB) 2,4,5-T
-2 920 1300 -4 1100 1600 -5 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 990 1200 -7 180\$ 0.50 -8 (0.01 0.70 (400\$) -9 (0.01 0.014 -1 380 770 -2 280 610 -3 (0.01 (0.01 -5 (0.01 (0.01 -5 (0.01 (0.01 -6 (0.01 (0.01 -1 (0.01	-1	800	<0.01
-4 1100 1500 1600 -5 1200 -6 1200 1200 1200 1200 -6 1200 2400 -7 0.18 0.26 (160%) 0.73 (460%) 0.77 (180%) 0.77 (180%) 0.70 (400%) 0.27 (180%) 0.70 (400%) 0.014 0.01 0.014 0.01 0.01 0.01 0.01 0.	-2	920	
-5 -6 -7 -7 -7			
-6	<b>-</b> 5		
-7 MS (0.05)	-6		
-74S (0.05) -714SD (0.05) -8 -9 -9 -0.01 -1 -1 -2 -3 -4 -5 -6 -7 -7 -7 -7 -7 -7 -7 -7 -7 -7 -7 -7 -7	<b>-</b> ?		
-7/4SD (0.05) -8 -9 -9 -9 -9 -9 -0.01 -0.014 -0.014 -0.01  21591-1MB -1 -2 -2 -3 -4 -4 -4 -5 -6 -6 -9 -0.01 -0.01  21484-2MB -2 -9 -0.01 -10 -10 -10 -11 -13 -13 -14 -15 -10 -10 -10 -10 -11 -13 -13 -14 -15 -17 -10 -10 -10 -10 -10 -10 -10 -10 -10 -10	-7MS (0.05)		
-8	-714SD (0.05)	0.20 (100%)	0.73 (460%)
21591-1MB	-8	(0.01 (180 <del>3</del> )	0.70 (400%)
21591-1MB -1 380 770 -2 280 610 -3 (0.01 (0.01 -4 (0.01 (0.01 -5 (0.01 (0.01) -6 (0.01 (0.01) 21484-2MB (0.01 (0.01) -9 (0.01 (0.01) -10 (0.01 (0.01) -11 (0.01 (0.01) -13 (20 (2.0  21413-1MB -1 (1.0 (0.01) -2 (0.01 (0.01) -2 (0.01 (0.01) -3 (0.01 (0.01) -1 (0.01) -1 (0.01) -2 (0.01 (0.01) -5 (0.01 (0.01) -7 (0.01 (0.01) -7 (0.01 (0.01) -8 (0.01 (0.01) -9	<b>-9</b>		
-1		(0.01	0.01
-1		<0.01	48.04
-2 -3 -4 -5 -6 -7 -7 -8 -9 -10 -11 -13 -14 -10 -10 -10 -10 -10 -10 -10 -10 -10 -10			
-3 -4 (0.01 -5 -6 (0.01 (0.01 (0.01 (0.01)	-2		
-4 -5 -6 (0.01	<b>-</b> 3		
-5 -6 (0.01 21484-2MB -2 -9 -10 -10 -11 -13 (0.01			
-6	<b>-5</b>		
21484-2MB	<b>-6</b>		
-2 -9 -10 -10 -11 -13 -13 -13 -1413-1MB -1 -1 -2 -2 -3 -5 -7 -8 -11 -11 -12 -13 -14 -15 -15 -16 -17 -17 -18 -17 -18 -19 -10 -10 -10 -10 -10 -10 -10 -10 -10 -10			10.01
-2 -9 -10 -10 -11 -13 -13 -13 -1413-1MB -1 -1 -1 -2 -2 -3 -5 -7 -8 -11 -11 -11 -2 -2 -3 -5 -7 -8 -11 -11 -11 -11 -11 -11 -11 -11 -11		<0.01	0.06
-9 -10 -10 (0.01 -11 (0.01			
-10 -11 -13 (0.01 (0.01 (0.01 (0.01 (0.01 (2.0  21413-1MB (0.01 (1.0 (0.01) (0.01 (0.01 (0.01) (0.01			
-11			0.94 (0.04
20			
21413-1MB	-15	<20	
-1	24.44.2. 4WD		12.0
-2 -3 <0.01 -5 -7 -8 -11 (7.0 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01			<b>&lt;</b> 0.01
-3			
-5 -0.01 -7 -8 -11 -0.01 -0.01 -0.01 -0.01 -0.01	-2		
-7 -8 -11 -11 -7 <0.01 <0.01 <0.01 <0.01	-5		
-8	- J - 7		
-11 (0.01 (0.01			
<b>4140</b>	- • •	0.02	0.06

#### Phenoxy Acids Resnalyses

21349-MB -MBS or MS 50 ppb 7RX -7MS (50 ppb) -7MSD (50 ppb)	<0.005 0.051 0.039 0.077 (76%) 0.0102 (130%)	<0.005 0.067 0.220 0.260 (80≴) 0.420 (400≴)
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1786

California Analytical Laboratories, Inc.

#### CREOSOTE

Creoscte (the 'coal tar' variety) is a mixture of polymuclear aromatic hydrocarbons used as biocide/biostat in the preservation of wood. Most of the principal components of the mixture are priority pollutants. We have examined both commercial creosote and the chromatogram in the reference cited in K. McKay's letter of December 23, 1985 and compared these data to the raw mass chromatograms raw Quan Lists for the samples IT-NCBC-R1-01 and IT-NCBC-There are a variety of polynuclear aromatic hydrocarbons present in the samples. Nevertheless, it is our conclusion that it is impossible to say that the creosote profile is present in the data. We base this conclusion on the low amounts of 2-methyl naphthalene and fluorene in one or both of the samples. Further, our own cressote data shows that phenanthrene dominates anthracene by a factor of greater than 6 to 1 in creosote. This does not occur in the cited samples. The data does not rule out the presence of creosote, but it is clear that the bulk of polynuclear aromatic hydrocarbons came from other hydrocarbon sources and further that the analysis for only "creosote" is not possible.

Summary of TCDD & TSP in Filter Samples

	6	<b>414</b> 5	ng/sample	ng/sample		
CAL Lab 1.D.	Number	U (sample)	Mess	Limit	$TSP (ug/m^3)$	Vol (m3)
21932-MB	819	1.00	QN	0.14		:
MBNS	Method Blank NS Y	1.00	13.6	;	;	;
-		1.00	2	0.59	18.6	4845.99
-5-	1132 Y	1.00	NO ON	0.27	44.9	4635.54
<b>K</b>	1133 Y	1.00	1.5	•	36.4	4845.99
4	1134 Y	1.00	QN	0.17	17.4	2877.06
	1135 Y	1.00	S	0.14	26.9	4547.10
-2-	1136 Y	1.00	ND	0.28	31.8	4637.07
- 2	1137 Y	1.00	QN	0.22	25.2	4547.10
7-	1138 Y	1.00	0.14	;	24.4	4721.23
٠.	1139 Y	1.00	S	0.13	41.9	1761.05
့်မှ	1140 Y	9.0	0.11	;	38.5	1596.16
	1141	1.00	1.1	;	55.7	1761.05
· φ <u>-</u>	1143 Y	1.00	NO ON	0.12	35.8	1828.50

V)

Report Date: 16-22-8

Lab: California Analytical Laboratories Case No. 21349 Batch/Shipment No.

Name	Sample C Number U	Aliquot C Wet Wt. U (grams)	PPB TCDD Meas	PPB TCDD Det. I	Inst 10	hate	Tine	320/ 322	332/ S 334 M	PPB Surg Meas X	Surrg X Acc <sup>1</sup> c	320	322	ន័	428*	332	, ¥	Š
10.51   260   26   26   26   2720.0   2779   2736.0   2779   2736.0   2770.0   277	¥	10.00	9	0.011	<b>6</b> 0	10/07/35	21:59:00			1.03	103				977032	1046270	1362800	
7 0.06 272	7	0.51	260		-	10/07/85	22:31:00	2.0	0.77 2	0.37	70	4233680	5354840	2650840	727050	774825	1004 100	
Y   50.00 ml   43.3   S   10/17/85   S   5   5   6   0.81   0.71   0.17   84   5356,000 657224.00   526884.00   4,0258   582448   F   10/07/85   S   2   3   3   0.77   5   10   5   5   5   5   5   5   5   5   5	<u>د</u> د	09.0	272		•0	10/07/85	22:53:00	6.70	0.79	67.5	86	5083820	7713790	3679450	799955	917779	1163060	
Y   0.61   236   236   236   236   2370   279   2.79   2	-03 Y	50.00 ml	43.3		<b>6</b> 0	10/17/85	15.15.00	0.81	0.71	0.17	-		65722490	~	440258	582468	823854	
1	-01 Y	19.0	236		80	10/07/85	Ç	2.0	0.78 1	16.39			/522380		546235	609775	778939	
10.19   NO   0.0076   10.077/85   23:50:00   0.78   0.77   17.74   103   4821660   6160990   27:0550   866648   17.71   17.74   10.19   NO   0.0043   Result from Tetra through Octa analysis   NO   0.007   NO   0.007   NO   0.007   NO   0.007	·01 Y	0.52	566		80	10/07/85		2.0	0.75	19.23	100	3709570	4698130	2311360	588306	877079	855600	
Y   10.19   ND   0.076   B   10/08/85   23:50:00   0.73   1.10   112   112   112   112   113	-01 Y	0.58	233		80	10/07/85	23:>0:00	9.78	0.77	17.74	103	4821660	6160990	2919550	809058	800048	1132170	
MO	1.02 Y	10.19	9	0.076	æ	10/08/85	23:50:00		0.73	1.10	112	•		•	167708	163716	22364.5	
NO   0.038   Result from Tetra through Octa analysis.   NO   0.036   Result from Tetra through Octa analysis.   NO   0.037   Result from Tetra through Octa analysis.   Result from Tetra through Octa ana	60-1		9	0.043	ž	esult from	Tetra thro	and Oct	a analy	sis.								
Y   50.00 ml   0.36   .   E   10/17/85   15:35:00   0.79   0.77   0.17   86   794464   1004730   390389   813564   1095490   19	1-09A		Ç <b>i</b>	0.038	ž		Tetra thro	ough Oct	a analy	/sis.								
No	1.04 Y	50.00 ml	0.36		బ	10/17/85		6.70	0.77	0.17	8		1004730	390389	813564	1095490	1419010	
NO   0.037   Result from Tetra through Octa analysis   NO   0.037   Result from Tetra through Octa analysis   NO   0.340   B   10/08/65   11:27:00   C.76   1.06   1.06   1.07   1.06   1.07   1.06   1.07   1.06   1.07	<b>~</b>		¥	9.079	×		Tetra thro	yugh Oct	a analy	sis.								
γ 10.07 NO 0.340 B 10/08/65 11:27:00	1 09		2		ž	esult from		Xugh Oct	(Jenal)	/sis.								
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10.01 ND 0.140 B 19/17/85 16:55:00 . 0.05 0.54 94	2.03 ₹	50.00 ml			•0	10/18/85			0.82	0.15		27890300	35784000	14925200	76659	99935	.21314	
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10.07 ND 9.060 8 10/08/65 13:19:00 · 0.80 0.99 91 · · · · 290802 366952 10.13 0.51 · 8 10/08/65 13:37:00 0.74 0.70 0.92 94 45672 61312 31316 187165 213156 ND 0.014 · Result from Tetra through Octa analysis.  ND 0.015 · Result from Tetra through Octa analysis.  ND 0.015 · Result from Tetra through Octa analysis.  50.00 ml 227 · 8 10/18/85 10:46:00 0.78 0.88 0.20 102 39956600 51048500 21022100 83684 96904	3.03 Y	\$0.00	250		80	10/18/85	11:46:00	9.3	0.87			60332100	77398900	31778300	102590	131960	152101	
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	S 04 Y	50.00 ml	257	•	80	10/18/85	10:46:00	0.78	83.0	0.20		39956600	51048500	21022100	83684	70696	109510	

650304 Ratio Unacc

468202

444955

135680

169312

171056

5

1118150 750541

883610 605151

741366 531377

804151

**398910** 

4369880 1806100

3497650 1421090

% ⊡

8 10/08/85 13:55:00 0.80 0.79 18.75 8 10/08/85 14:20:00 0.79 0.81 18.74 Result from Tetra through Octa analysis. 8 10/22/85 12:17:00 1.01 0.72 1.01

Result from Tetra through Octa analysis Result from Tetra through Octa analysis.

0.038 0.78 0.013 0.018

55555

21591-1RX MUNCBC-R1-01 Y 0.51 21591-2RX HUNCBC-R2-01 Y 0.54 21591-3 MUNCBC-R2-02 21591-4RX HUNCBC-R2-03 Y 10.00 21591-5 HUNCBC-R2-09A (19:00) 21591-6 HUNCBC-R2-05A (19:40)

MB = Method Blank
P = Partial Scav/Confirmatory Analysis
NS = Mative TCD0 Spite
D = Duplicate/Fortified Field Blank
R1 = Re-injection

#### APPENDIX T

CLEOSOTE CHROMATOGRAM SUPPLIED BY SUPELCO, INC.

## Stable Phases for Separating High Boiling Mixtures

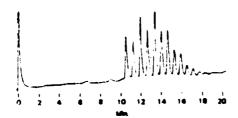
arations of very high boiling mixtures glycerides, cholesteryl esters, etc.) require a stationary phase with good stability at temperatures above 300°C.

For packed columns, Dexsit® 300, a low polarity carborane silicone, can be used at up to 450°C with negligible bleed. Short (12° to 18") columns of 1% Dexsil are recommended for many types of samples (Figures A - D). For complex hydrocarbon mixtures, 3% Dexsil is suggested (Figure E). For more details, request Bulletins 743 and 758.

SPB-1 capillary columns (bonded SE-30 phase) offer a stable baseline and thermal stability to 320°C. We recommend you use a 0.75mm ID column and on-column injection (flash vaporization) when analyzing high boiling mixtures. This will prevent the discrimination that occurs with splitter systems. Under these conditions, branched hydrocarbons (short peaks) and n-alkanes (tall peaks) in a wax sample are well separated (Figure F). You can easily install and use a 0.75mm ID column in a packed column GC (see "Wide Bore Capillary Columns" in the index or request Bulletin 814 for more details).

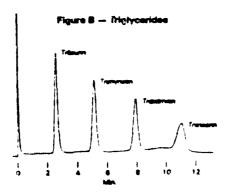
Floure A - Pentserythritol Esters

間にどころ



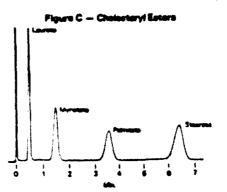
1% Jessé 300 on 100/120 Supercoord 18" x 1/8" SS. Col Temp. 125" to 300°C at 8°C min. Inset Temp. 325°C, Det Temp. 350°C, Flux Rate. 20m $\nu$ min. N<sub>p</sub>. Sample 1  $\mu$ l chloroform containing 10  $\mu$ g esters.

Packing: Cat. No. 1-1972. \$81/20g



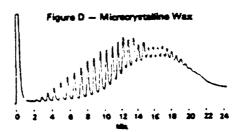
1% Deadd 300 on 100/120 Supercoport, 18" r 1/8" SS. Col. Temp. 275" to 350°C at 8°C/min, limit Temp. 325°C, Det. Temp. 350°C, Flow Rate 20mi/min, N<sub>3</sub> Sample 1 $\mu$ I chloroform containing 1 $\mu$ g each snghrounds.

Packing: Cat. No. 1-1972, \$81/20g



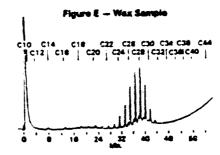
1% Dexad 300 on 100/120 Supelcapert, 18" x 2mm iD glass, Col. Temp. 300" is 350°C at 6°C/min., Intel Temp. 350°C, Flow Rate. 40mir/min., N<sub>2</sub> Sample. 1<sub>pl</sub> chloreform containing 1<sub>pl</sub> sech exter.

Packing: Cat No 1-1972, \$81/20g



1% Jexad 300 on 100/120 Supelcosent, 18" x 1/8" SS, Col. Temp. 175" to 350°C at 8°C/mm, litter 100 pc. 150°C, Plow Rate 20m/mm, N, Det FIO Sers. 16 x 10 " AFS, Samots 1  $\mu$ I chloroform, containing 30 $\mu$ g wex.

Packing: Cat. No. 1-1972. \$81/20g

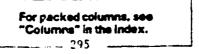


3% Dersid 300 on 100/126 Supercoport, 6" x 1/8" 35, Col. Temp... 100" to 380"C at 4"C/min., Flow Rate 20mil/min., N<sub>p.</sub> Semple... 1<sub>jul.</sub> chloroforms. containing 30 up with

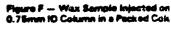
Packing: Cat. No. 1-1973, \$107/20g

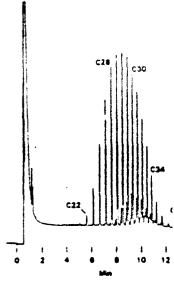
Additional Packing for High Boiling Aromatics

1-2132 10% SP-2250 on 100/120 Supelcoport, 20g \$62



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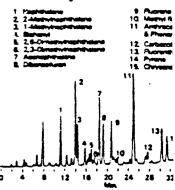
SPB-1 wide bore capillary column, 30m x 0 1 0 µm film, Col. Temp., hold 2 min at 130 320°C at 18°C-min, and hold 15 min. Temp 350°C, Flow Rate. 15cc/min, He trolledt, Det.: FID, Sens.: 4 x 10°19 AFS, Sen a convinences wax in undecane, 130 µg/µL (

Column: Cat. No. 2-3755,

#### **High Boiling Aromatics**

We recommend 10% SP-2100 silicone for separating high boil matics (Figure G). A 10% S methyl phenyl silicone is also us such separations. For more deliquest Bulletín 743.

Figure G - Creceote



10% SP-2100 on 100/120 Supelcoport, 1 SS, Col. Temp., 100° to 300°C at 6°C/min , 20mi/min , N<sub>p.</sub> Det., FID, Sample, 0.1 μL

Packing: Cat. No. 1-1989, \$

#### APPENDIX U

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BATTELLE COLUMBUS LABORATORIES ANALYTICAL METHODOLOGY AND RESULTS FOR DIOXIN/FURANS IN NCBC SAMPLES

#### APPENDIX U

# BATTELLE COLUMBUS LABORATORIES ANALYTICAL METHODOLOGY AND RESULTS FOR DIOXIN/FURANS IN NCBC SAMPLES

#### Introduction

This appen'ix describes the analytical procedures used to determine the levels of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in three soil samples and three QA/QC samples submitted by EC&C Idaho, Inc. One of these soil samples, HU-NCBC-R2-02, was treated soil from the Huber NCBC test. The other two soil samples were from the IT Corporation testing NCBC, which was concurrent with the Huber testing. These data are included because of the QA/QC program interactions. The specific levels of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) and 2,3,7,8-tetrachlorodibenzofurans (2,3,7,8-TCDF) as well as the congener class concentrations of the tetrachloro- through octachloro- PCDD/PCDF classes were determined.

#### Extraction

Ten grams of each of the three samples, as well as the duplicate and native spike samples, were weighed and transferred to Soxhlet extractors. Sample number 1T-NCBC-R1-01 was used as the duplicate, and HU-NCBC-R2-02 was used as the native spike. These five samples and the method blank were spiked with 25.0 ng each of three isotopically labelled internal standards: 2,3,7,8-tetrachlorodibenzo-p-dioxin- $^{13}$ C<sub>12</sub> (2,3,7,8-TCDD- $^{13}$ C<sub>12</sub>) 2,3,7,8-tetrachlorodibenzo-furan- $^{13}$ C<sub>12</sub>

a. Information is referenced from Battelle Columbus Laboratories "Final Report on Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Soil Samples, prepared for EG&G Idaho, Inc., dated May 21, 1986.

 $(2.3.7.8-TCDF^{-13}C_{12})$ , and octachlorodibenzo-p-dioxin- $^{13}C_{12}$  (OCDD- $^{13}C_{12}$ ). The samples were then Soxhlet-extracted for 18 hours using benzene.

#### Extract Cleanup

The benzene extracts were concentrated to approximately 5 ml using three stage Snyder columns, diluted with 5 ml of hexane, and transferred to multilayered columns containing activated silica gel, 44 percent concentrated sulfuric acid on silica gel, and 33 percent 1M sodium hydroxide on silica gel. The columns were rinsed with 70 ml of hexane and the entire eluates were collected. The purpose of these columns was to remove acidic and basic compounds from the extracts as well as oxidizable materials.

The benzene/hexane eluates were concentrated using a gentle stream of nitrogen gas and solvent-exchanged into hexane. The hexane solutions were chromatographed through columns containing approximately 1 gm of activated basic alumina using hexane/methylene chloride (97:3, v/v), and hexane/methylene chloride 1:1, v/v) as elution solvents. The 1:1 hexane/methylene chloride eluates were collected, concentrated to near dryness, and dissolved in 20  $\mu$ t of n-decane containing 10 ng of 1,2,3,4-TCDD- $^{13}$ C<sub>12</sub>, which was used as an absolute recovery standard. The solutions were stored at 0°C and protected from light until analyzed.

#### Analysis

The extracts were analyzed and quantified for PCDD/PCDF using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The KRGC/HRMS consisted of a Carlo Erba Model 4160 gas chromatograph interfaced directly into the ion source of a VG Model 7070 mass spectrometer. The chromatographic column was a 60M DB-5 fused silica column using helium carrier gas at a flow velocity of 25 cm/sec. The mass spectrometer was operated in the electron impact (EI) ionization mode at a mass resolution of 9,000-12,000 (M/AM, 10 percent valley definition).

The operating parameters of the HRGC/HRMS are summer ed in Table U-1. All HRGC/HRMS data were acquired by multiple in detection (MID) using a VG Model 2035 Data System. The exact masse in nitored are shown in Table U-2.

#### Quality Assurance

The operation of the HRGC/HRMS was evaluated each day by analyzing standard mixtures of PCDD/PCDF isomers. These consisted of native and isotopically labelled isomer mixtures used to determine response factors, mixtures of selected PCDD/PCDF isomers to evaluate the stability of the chromatographic elution windows, the TCDD isomer mixtures to evaluate isomer resolution. The mass accuracy of the MID unit was evaluated at least every 4 hours by focusing selected ion masses from perfluorokerosene (PFK) and correcting the slope to account for minor variations. Mass focus stability was assured by the use of a reference PKF "lock mass" to correct for any mass focus drift.

A method blank and a native spiked sample were processed during the extraction and cleanup of the samples. The results of these analyses are summarized in Table U-3. The raw mass spectral data, areas and heights, are presented in Table U-4. The method blank was free from PCDD/PCDF contamination except for trace levels of HpCDD and OCDD. Background levels of higher chlorinated dioxins are periodically observed in low level PCDD/PCDF analyses. The average native spike recoveries were within approximately 6.5 percent of the spiked value.

#### Quantification

The PCDF/PCDD isomers were quantified by comparing the sum of the two ion masses monitored for each class to the sum of the two ion masses monitored for the corresponding internal standard. The  $2,3,7,8\text{-TCDF}\text{-}^{13}\text{C}_{12}^{-\frac{1}{12}}\text{ was used to quantify the tetrachlorodibenzodioxins}$ 

<sup>\*</sup> was used to quantify the tretrachlorodibenzofuran issues, and 2,3,7,8,-TCDD-  $^{13}\mathrm{C}_{12}$ 

OCDD-<sup>13</sup>C<sub>12</sub> was used for the heptachloro- and octachlorodioxins and furans. Experimental relative response factors were obtained by analyzing a test mixture that contained representatives of the tetrachloro- through octachloro- PCDD/PCDF classes. These response factors were included in all calculations used to quantify the data. The response factors were calculated by comparing the sum of the areas of the two ion masses monitored for each congener class to the corresponding internal standard ion masses. The experimental response factors were:

	Data	File	
Analyte	591018	591109	Average
TCDF	1.0620	0.9982	1.0301
TCDD	1.0270	1.1209	1.0740
PCDF	0.9463	1.0413	0.9938
PCDD	0.3942	0.4622	0.4282
HxCDF	0.9669	1.0277	0.9973
HxCDD	0.4425	0.4893	0.4659
HpCDF	2.0288	2.4330	2.2309
HpCDD	3.054_	3.0823	3.0682
OCDF	0.9205	1.0532	0.9869
OCDD	1.0568	1.0776	1.0672

Internal Standard RRF Relative to  ${\rm 1.2,3,4\text{-}TCDD}^{13}{\rm C}_{12} \ \ {\rm for} \ \ {\rm Recovery} \ \ {\rm Calculations}$ 

TCDF-13C12	0.9695
TCDD-13C12	1.1-49
OCDD-12C12	0.2491

The formula used for quantifying the PCDD/PCDF iscmers was:

Level ppb) = Areas of Quantification Masses x Amount of Internal Standard (ng)
Areas of Internal Std. Masses x Resp. Factor x Wt. Sample (g)

The criteria used to identify PCDD and PCDF isomers were:

- (1) Simultaneous responses at both masses
- (2) Chlorine isotope ratios within ±15 percent of the theoretical values
- (3) Chromatographic retention times within windows determined by analyses of standards
- (4) Signal-to-noise ratio equal to or greater than 2.5 to 1.

The 2,3,7,8-TCDF/TCDD isomers included the additional criterion that they elute within ±2 seconds of their isotopically labeled analogs. A limit of detection was calculated for samples in which a particular chlorination class was not detected. The formula used was:

Limit of Detection (ppb) =  $\frac{\text{Hts of Quant. Masses x Amt. Int. Std. (ng) x 2.5}}{\text{Hts. of Int. Std. Masses x Resp. Factor x Wt. Sample (g)}}$ 

#### Results

WASHESS NICESCO BITTON TOSSINI MESSINI MESSINI PERSINI PERSINI MESSINI

The levels of PCDD/PCDF determined in the samples are summarized in Table U-3. Analysis of sample number IT-NCBC-R1-01 in duplicate indicated the presence of 2,3,7,8-TCDD at the levels of 165 ppb and 170 ppb. Since the level of native 2,3,7,8-TCDD in the sample is approximately 70 times higher than the level of the internal standard, it is possible that the response of the ion source of the mass spectrometer was not linear. The sample was reinjected with a 0.1  $\mu$ l injection size (rather than 2.0  $\mu$ l). This analysis indicated the level of 2,3,7,8-TCDD to be 220 ppb; however, it is possible that the actual level is even higher. In an attempt to obtain a more representative value for the level of native

material in this sample, 100 ppb of 2,3,7,8-tetrachlorodibenzo-p-dioxin-<sup>37</sup>Cl<sub>4</sub> (2,3,7,8-TCDD<sup>37</sup>Cl<sub>4</sub> was spiked into the sampla extract. The amount of 2,3,7,8-TCDD<sup>37</sup>Cl<sub>4</sub> was then determined relative to 2,3,7,8-TCDD<sup>13</sup>C<sub>12</sub>. The sample was diluted by a factor of ten, and the native 2,3,7,8-TCDD was quantified based on the 2,3,7,8-TCDD-<sup>37</sup>Cl<sub>4</sub>. As in the 0.1 µl injection of the nondiluted sample, this set of analysis indicated the level of native material to be 220 ppb. The formulas used for this calculation were:

Stnd R<sub>f</sub> 
$$^{13}C_{12}/^{37}C_{14} = \frac{\text{Area} \ ^{13}C_{12} \ \text{Quant Masses in Stnd x Amt.}}{\text{Area} \ ^{13}C_{12} \ \text{Quant Masses in Stnd x Amt.}} \frac{^{37}C_{12} \ \text{(ng)}}{^{13}C_{12} \ \text{(ng)}}$$

and

1, (Stnd 
$$R_f^{13}C_{12}^{37}C_{4}$$

Table U-4 lists the area data and the concentrations used in these calculations. The reconstructed ion chromatograms for these three data files (Stnd, Lower Sen: itivity, High Sensitivity) are contained in the section of this report that is labeled "IT-NCBC-R1-01<sup>37</sup>C1<sub>4</sub>-Spike". The only alternative available to obtain a reliable value for the amount of native material in this sample involves the additional cost of repeating in extraction, using a smaller sample size (1 gram versus 10 grams). Because of cost and time constraints, it was decided that it be inappropriate to proceed any further with this sample.

Sample R<sub>f</sub> 
$$^{13}C_{12}^{/37}C\ell_{4} = \frac{\text{Area} \ ^{13}C_{12} \ \text{Quant Masses is Stnd X Amt.} \ ^{37}C\ell_{4} \ \text{(ng)}}{\text{Area} \ ^{37}C\ell_{4} \ \text{Quant Masses x Amt.} \ ^{13}Cl_{12}^{/} \ \text{(ng)}}$$

For those samples in which a particular chlorination class was not detected, a detection in parts-per-billion (ppb) is listed in Table U-3. The height and area data used to calculate the concentrations and detection limits can be found in Table U-5.

The percent recoveries of the internal standards in each sample and the chlorine isotope ratios for isomers or isomer classes detected are reported in Table U-6 and U-7, respectively. The formula used to calculate the percent recoveries was:

Percent Recovery = 
$$\frac{\text{(Area of Quant. Masses of Stnd)} \times \text{(Amt. 1,2,3,4,-TCDD-}^{13}\text{C}_{12}) \times \text{100}}{\text{(Area of Quant. Masses of 1,2,3,4-TCDD-}^{13}\text{C}_{12}) \times \text{(Amt. Stnd)} \times \text{R}_{f}}$$

where  $R_f$  is relative to 1,2,3,4-TCDD- $^{13}C_{12}$ .

#### Single Ion Current Chromatograms

The single ion current chromatograms for the samples, standards, and decane analyses are included in the referenced Battelle report (see footenote at beginning of this appendix). They were assembled in analysis order and are cross-referenced by the table that prefaces each section. The data files are six-digit numbers, with the first two numbers denoting the instrument logbook in which the analysis is recorded. The third and fourth numbers indicate the package in the logbook, and the fifth and sixth numbers mark the line on which the entry was made. All information pertaining to extraction and workup of the samples can be found in Battelle Laboratory Book Number 40196. The GC/MS acquisition parameters can be found in Laboratory Record Book Number 41270.

#### APPENDIX V

CAL EP TOXICITY TEST RESULTS FOR HUBER TEST BAGHOUSE FILTER MATERIAL



## California Analytical Laboratories, Inc. \$2544 Industrial Baulevard • West Sacramento, CR 95691 • (916) 372-1393

January 31, 1986 Lab No. 21591-A

Kathy MacKay
EG & G Idaho, Inc.
1955 Fremont Ave.
Idaho Falls, Idaho 83415

One soil sample was re-submitted for metals analysis by EP Toxicity Test Method (EPA 1310).

CAL I.D. 21591-4

#### RESULTS:

Analytical and quality assurance results are presented in the attached data sheets. Spike recoveries for post-digestion spike are low for some elements. This may be attributed to the high initial sample pH (11.9). The maximum allowable volume of 0.5N acetic acid to be added to a sample is 4 milliliters per gram according to Method 1310. In this sample, a final pH of 9.7 was achieved after addition of 8 milliliters acid to a 2 gram sample. This pH is too high for dissolution of some elements.

Paul Taylor

President

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Conna Gilmore

Manager Inorganic Services

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#### EF TOXICITY

#### DATA SHEET

SAMPLE I.D.: HU-NCBC-R2-03

CAL I.D.: 21591-4

Metals Arsenic	mg/L Leachate Regulatory Limit 5.0	mg/L Found in Leachate <1.0
Barium	100	0.75
Cadmium	1.0	<0.1
Chromium	5.0	<0.1
Mercury	0.2	<0.01
Lead	5.0	1.1
Selenium	1.0	<0.5
Silver	5.0	<0.5

PREPARED BY:

APPROVED BY:

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California Analytical Laboratories, Inc.

# EP TOXICITY TEST QUALITY ASSURANCE DATA SHEET

POST-DIGESTION

SAMPLE I.D.: HU-NC BC-R2-03

CAL I.D. 21591-4

				Po	at Diges	tion Sp	ike '
			Dup.	Rel.	mg/	L	_
Metals Arsenic	mg/L Leachate Regulatory Limit 5.0	Sample mg/L <1.0	Sample mg/L <1.0	Diff.	Spike Result 1.85	Spike Level 2.00	Recove
Barium	100	0.75	0.68	9.8	2.24	1.50	99
Cadmium	1.0	<0.1	<0.1	0	0.214	0.200	107
Chromium	5.0	<0.1	<0.1	0	0.191	0.250	76
Mercury	0.2	<0.01	<0.01	0	2.10	2.0	105
Lead	5.0	1.1	1.7	43	3.03	2.00	96
Selenium	1.0	<0.5	<0.5	0	0.420	1.00	42
Silver	, 5•0	<0.5	<0.5	0	0.063	1.00	6.3

PREPARED BY: RB

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California Analytical Laboratories, Inc.

## APPENDIX W

BATTELLE COLUMBUS LABORATORIES PRIORITY POLLUTANT METALS AND CYANIDE ANALYTICAL RESULTS FOR SIX NCBC SOIL SAMPLES

#### APPENDIX W

BATTELLE COLUMBUS LABORATORIES PRIORITY POLLUTANT METALS AND CYANIDE ANALYTICAL RESULTS FOR SIX NCBC SOIL SAMPLES

Ag, As, Be, Cd, Cr, Cu, Ni, Pb, Se, Ti & Zn Analysis

A 5.0 g sample was weighed from the thoroughly mixed content of the jar containing the original sample. The sample was placed in a 150 mL and 5.0 ml of concentrated HNO, were added to it. The beaker was placed on a hot plate set at approximately 90°C and left to digest for 3 hours without allowing it to boil. The beaker contents were then evaporated to near dryness prior to the addition of 5.0 mL of concentrated HNO3. The beaker was placed back on the hot plate of the contents were evaporated to near dryness again. This step was repeated three times after which 5 mL of 1:1  $\mathrm{HNO}_3:\mathrm{DI}\ \mathrm{H}_2\mathrm{O}$  and 7.5 mL DI water were added. The mixture was heated for 15 minutes prior to filtering through white ribbon paper (No. 584) with pulp into a 50 mL volumetric flask. The contents of the volumetric flask were brought to volume using DI water. This solution was analyzed using either Zeeman graphite furnace atomic absorption spectrophotometry (Z-GF-AAS) or inductively coupled argon plasma (ICAP) according to the instructions given in the manufacturer's manual and the wave lengths given in the attached Tables 3 & 4.

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## Hg Analysis

A 1.0 g sample was weighed from the thoroughly mixed contents of the jar containing the original sample. The sample was then placed in a 125 mL Extended from the flask and to it 10 mL aqua regia solution were added. The flask was placed on a steam bath and heated for approximately 1 hour then diluted to 50 mL with DI water. 2.5 mL concentrated  $\rm H_2SO_4$ , 10 mL KMnO\_4, and 4 mL  $\rm K_2S_2O_8$  were added. This solution was heated in a sceam bath for approximately 4 hours\* were added prior to analysis by cold vapor atomic

<sup>\*</sup> After which I drop of hydroxylamine hydrochloride and 5 mL stannous chloride.

absorption spectrophotometry (CV-AAS) at the wavelength given in the attached Table 3.

## Sb Analysis

A 5.0 g sample was weighed from the thoroughly mixed contents of the jar containing the original sample. The sample was placed in a 150 mL beaker and 10 mL aqua regia were added to it. The mixture was placed on a hot plate set at approximately 90°C and left to digest for three hours. The beaker was removed, cooled and its contents filtered into a 100 mL volumetric flask through a white ribbon (No. 589) paper with pulp. The beaker and the filter were washed with DI water and the washings were added to the volumetric flask DI water was used to bring the volume to 100 mL prior to analysis by Zeem graphite furnace atomic absorption spectrophotometry (Z-GF-AAS) at the wave length given in the attached Table 3.

## QA/QC

All the samples were logged into the central laboratory record system and given Battelle numbers prior to distribution to the individual analysts. All the laboratory activities and results were recorded in Laboratory Book No.'s 40602 and 39818. Sample 40196 53-2 was used for both duplicate and spike recovery studies. The spiking was accomplished by adding the spike level given in Table 2 to the soil sample immediately after adding the acid to it. The spiked sample was then taken through the sample analytical procedure as for the unspiked sample.

# APPENDIX X

DISCUSSION ON SOIL PRETREATMENT FACILITY

#### DISCUSSION ON SOIL PRETREATMENT FACILITY

The soil pretreatment facility can prepare a variety of contaminated soils for treatment in the AER. The dry system is capable of crushing, blending, drying, pulverizing, and sizing a variety of composite soils, of sizes up to 10 inches, to produce a ground, free-flowing material that will pass through a 35-mesh screen, thereby assuring the prepared soil will have the desired AER retention time.

Figure X-1 presents the detailed flow diagram for the soils pretreatment facility. Figure X-2 shows the general arrangement plans and key views to aid in understanding the following discussion of the different phases.

## Soils Receiving and Handling

All solid feed material is delivered to the soil pretreatment facility in sealed, portable containers (bins). Each bin is designed to hold approximately 12 cubic yards or 30,000 lb of material.

Plant operators will use a recently loaded bin or will take a loaded bin out of the bin staging area with a roll-off truck or forklift and place them into a 40,000-lb capacity feed surge bin.

As the bin is positioned exactly over the feed chute, a hydraulic mechanism is actuated to gently lower the bin into the feed opening. The feed chute has peripheral gasketing to prevent any dust leakage.

The feed bin has a stationary 10-inch grid grizzly located under the feed chute to divert oversize material to a gravity chute, which will collect occasional trash solids in a tote bin. The grizzly oversize material will be removed and handled separately.

A 42-inch-wide bucket elevator feeder is installed under the feed bin inside an enclosed housing, which is vented to the primary bag filter. This feeder has a variable-speed drive, which can be adjusted to match the feed rate established by the operator in the control room. The bucket elevator discharges onto a 30-inch feeder belt with a self-cleaning, cross-belt, electromagnetic tramp iron magnet mounted over the 30-inch feeder belt to remove magnetic metal objects to a separate chute and tote bin. A metal detector is installed downstream from the tramp iron magnet to sound an alarm and stop the weight belt if nonmagnetic metal appears on the 30-inch belt. A manually operated hydraulic picker mechanism pushes the metal or other objects off the belt to another chute and tote bin, which collects this material for disposal by other means. The 30-inch belt gravity feeds the primary crusher.

From this point in the process, all solid materials are handled either by gravity flow between process equipment or by dense-phase pneumatic solids transport systems, which convey fine dry material to the next process step.

## Soils Crushing, Drying, and Pulverizing

The feed to the process can be a maximum of 10 inches diameter into the primary crusher, which is a 10-hp impact breaker, having the capability to open circuit crush the incoming feed at the rate of 6,700 lb/hr to a 1-1/2-inch maximum particle size. This crushed product is then fed by gravity into the indirect-heated dryer.

The primary crusher can process feed soil mixtures cont ining as high as 25-percent moisture, but second-stage comminution in hammer mill and screen separations at 35 mesh are not possible with solids containing more than about 1-percent moisture. Therefore, the material is dried ahead of the hammer mill. The process drying system consists of a disc dryer, to dry 30-percent moi cure feed soil to 1-percent moisture at a feed rate of 6700 lb/hr. The dryer solids at 210°F gravity discharge into the hammer

mill feed bin. The indirect-heated dryer is fed by a gravity discharge from the primary crusher located above the dryer in Module 02.

The heat for the dryer system comes from a package gas-fired Dowtherm system with 3.0-million-Btu/hr heat capacity. The unit is equipped with a 1-1/2-hp blower and 20-hp pump. This dryer will have its torus disc flights hard-surfaced to resist the wear from the 1-1/2-inch-solids in the feed soil.

The hammer mill operates in closed circuit with a multideck vibrating screen to produce 6000 lb/hr of -35 mesh product. An approximate 150 percent circulating lead of +35 mesh solids recirculates back to the hammer mill to achieve the fineness of grind required for the process specification. This is achieved by gravity feeding the 1-1/2-inch-sized dryer discharge into the hammer mill reed bin where a gravity discharge chute from the screen located in the module above the dryer drops about 8900 lb/hr of +35 mesh screen oversize product into the hammer mill surge bin. The combined flow passes through a rotary valve and into the hammer mill, which can produce pulverized material that is 99 percent -1/4 inch with an average particle size of about 16 mesh with 40 percent by weight of -35 mesh material.

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The hammer mill discharge is fed to the dense-phase pneumatic conveyor system, which elevates the coarse ground product to feed the vibrating screen. The system could accumulate a circulating load of lightweight fibrous material resulting from shredding wood or cloth in the mill. This material is intermittently passed through a fiber chopper, which drops the fibrous material at the rate of 100 lb/hr into a very fine powder. This material is gravity-dropped into a surge bin that also collects -35 mesh screen product and dust from the bag filters.

The surge bin discharges into a rotary valve, which feeds the dense-phase pneumatic transport system. This system conveys the comminuted product at and approximate rate of 6,000 lb/hr to its bag filter, which has been modified to provide a 10,000-lb surge capacity in the bottom of the bag filter. The product surge bin feeds a loss-in-weight feed system that

gravimetrically controls the rate of feed to the AER in the demonstration plant. The inlet flank to the AER is located 80 feet above the ground. The AER is fed through a rotary valve capable of discharging against the 1 psig pressure in the AER.

## Dryer Off-Gas and Plant Ventilation Systems

The water vapor, air, and dust leaving the dryer vent are heated electrically, if necessary, and conducted to a separate bag filter. A backup bag filter is installed in the off-gas line to intercept any solids flow in the event of a fabric bag failure. Vapors entrained with the air and water vapor are swept out of the dryer into the vent gas ductwork with an ID fan and are captured in five carbon adsorption filters.

To prevent fugitive emissions from the facility, a complete plant ventilation system is provided to allow every bin, crusher, screen, and pneumatic conveyor to have an outlet into a bag filter system. Motive force is provided by a 3500-scfm, 25-hp, ID fan.

The paretreatment facility will receive 13.8 kV line power from outside the battery limits to feed a 500 kVA transformer. This transformer supplies 400-V, 3-phase\* motor starter modules, a 480-V, 5-k kVA transformer for one-phase lighting power, a 50-a, 30-pole lighting receptable panel, a 20-a, 12-pole instrument panel, and a 60-a welding receptable. All electrical switchgear is housed in a single module.

## Description of Modules

The pretreatment facility equipment has been arranged so that it can be fitted into 11 modules, each 12 feet wide by 11 feet 2 inches high by 39 feet long, with a maximum weight of 72,000 lb (i.e., module steel plus empty equipment). These dimensions and weight were chosen to allow for unrestricted transport on lowboys by road throughout the U.S. The modules are identified as follows:

Thower to a motor control center containing fifty 445-V, 3-phase.

- Module 01 Feed surge bin, 42-inch bucket elevator, 30-inch belt feeder, grizzly, hopper, and 5-ton electric hoist.
- Module 02 30-inch belt feeder, tramp iron magnet, picker, and primary crusher.
- Module 03 Dryer, Dowtherm unit. and hammer mill feed bid.
- Module 04 Hammer mill, flexible screw conveyor, and rotary valve.
- Module 05 Vibrating screen, fiber chopper, knifegate, collection bin, rotary valve, and automatic sampler.
- Module 06 Plant ventilation primary bag filter, dust leak detector, electric air heaters, and screan air conveyor.
- Module 07 Dryer primary bag filter, dry secondary bag filter, dust leak detector, air heaters, blower, and activatea carbon adsorbers.
- Module 08 Plant ventilation secondary bag filter, electric air heater, and blower.
- Module 09A 500-kVA transformer, 5-kVA transformer and motor control center circuit breakers, motor starters, and lighting and instrument panels.
- Module 09B Stairways.

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Medule 21 Product air conveyor, product surge bin, and product feeder.

The first four modules in the process flow are stacked to achieve gravity transfer of material between equipment. Screw conveyors, or chain conveyors have not been used, as these would present added cleanup

difficulties. Belt feeders are used at the reed end for process feed control and to provide for tramp metal removal and pickup. Any dribble from these two feeders will be collected in tote hims for disposal. Elevation of material is by two air conveyor systems, one for the recycle load between the secondary crusher and screen and one for the product feed to the reactors.

A monorail/hoist installed in the top module will be used for raising and lowering bins containing grizzly oversize, tramp iron, etc., through a hoistway

Heating and cooling of the electrical control center during winter and summer will be a 2-ton unit heat pump. The temperatures in the module will be maintained between 40°F minimum and 90°F maximum. Siding will be provide for the modules to shield against wind during maintenance and to provide security

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# APPENDIX Y

DETAILED COST ESTIMATE FOR AER TREATMENT AND CLEANUP

APPENDIX Y, CAMIBIL 1. OFTAILED COST ESTIMATE FOR AET TREATMENT OF 20,000 TONS OF SUIL (REFERENCE CASE)

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Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
CATEGORY 1: COMMON REMEDIAL COSTS					
MOBILIZATIGA					
General mobilization and		1.5	000,56	95	5% of Category I subtotal cost
Trial burn Community relations		25	150,000 40,000	150	
CONSTRUCTION					
Ancillary buildings and					
equipment.  a) Water treatment facility	-	1.5	300,000	300	GAL facility, 25 gpm (50 gpm peak)
<ul><li>b) Office trailer</li><li>c) Employee trailer -</li></ul>	12	<b>9 W</b>	380 380	vr. vn	for decon, runoff, quench 50 ft x 10 ft trailer 50 ft x 10 ft trailer
oreakroom d) Forklift for material preparation	12	<b>9</b>	1,581	61	;
Utility upgrade: a) Electrical services	~	rs	000*89	89	\$58,000 for main facility, \$10,000
_	<b>د</b>	EA LS	1,400 20,000	20	for excavation site area
d) Sewer connection e) Natural qas line	<b></b> ,	rs rs	30,000 30,000	22	Fuel to heat soil driver
_		rs	10,000	20	
Excavation site buildings:  a) Decontamination trailer reports (2)	28	8	000*1	58	;
b) Vehicle decontamination station	_	57	30,000	30	•

APPENDIX Y, EXHIBIT 1. (continued)

Comments			for daily operation logal storage for 7 days of operation logal storage for 7 days of operation for 7 days holding	or 220 days or 220 days or 220 days for each	rr 220 days	8 hr/day for 220 days each worker 25% of H&S personnel cost 	Purchase and disposal of protective equipment Purchase and disposal of protective equipment for truck drivers
		::	3 for daily operating for 7 days of operating yd3 net vol, to 12 yd3 net vol, to for 7 days holding	8 hr/day for 220 days 8 hr/day for 220 days 8 hr/day for 220 days driver	8 hr/day to	8 hr/day fo 25% of H&S 	Purchase and disposa protective equipment Purchase and disposa protective equipment drivers
Cost (\$1000)		85 36	48 15 140 140	46 46 92	<del>2</del>	88 22 10 9	12
Unit Price		7,100	1,984 5,000 5,000 5,000	5 5 5 7 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	17	25 100 600 1,100	56 26
Units		M M	E P P S	# # # # #	Ě	EA EA	Q Q 9
Quantity		12	24 3 28 28	1,760 1,760 3,520	09/*1	3,520 220 16 8	1,320
ltem	EXCAVATION LOAD AND HAUL	Equipment:     a) Backhoe rental     b) Tractor (with hoe ram)	rental c) Rolloff truck rental (2) d) Rolloff boxes for operations e) Rolloff boxes for untreated soil storage f) Rolloff boxes for treated soil storage	Labor:  a) Backhoe operator b) Tractor operator c) Rolloff truck drivers (2)	d) Excavazion foreman HEALTH AND SAFETY	Health and safety personnel (2) Per diem and general expense for H&S personnel Physicals (2/worker)	Level C (6 sets)Level D (2 sets)

APPENDIX Y, EXHIBIT 1. (continued)

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ltem	Quantity	Units	Unit Price	Cost (\$1000)	Comnents
SITE RESTORATION					
Spreading treated soil: a) Dump truck b) Truck driver	12	<b>9</b> £	1,984	24 46	8 hr/day for 220 days
Regrade site	84,700	λS	0.81	69	
Topsoil placement	2,820	رخ	13.50	88	15% of 18,800 cy
Erosion matting	84,700	λ	08.0	89	;
Seeding and revegetation Salvage of rolloff boxes	84,700 59	S.K.	1.40 -2,500	-148	50% of cost, 3 operations, 28 untreated soil, and 28 treated soil
CUMMUN REMEDIAL COST SUBTOTAL				1,997	
Contingencies (25%) General administration (13%)	: :	::	;;	499	Factor applied to subtotal Factor applied to subtotal and
Contractor fee (8%)	;	;	:	3,046	contingencies Factor applies to subtotal and contingencies and GA
CATEGORY 1: TOTAL COMMON REMEDIAL COST	0S1				
CATEGORY 2: COMMON OPERATING AND MONITORING COSTS	NITORING CO	2150			
GENFRAL					
On-scene coordinator Per diem and general expense	12 330	M0 0 ¥	5,500 50	99	\$25/hr x 8 hr/d x 27.5 d/mo 25% of labor cost
MONITORING AND ANALYSIS PROGRAM					
On-site lab facility	-	rs	250,000	250	Includes enclosure, high resolution GC/MS, ancilliary enument
Analytical Lab Operation:	48	C	8.333	400	
To dies and coness overce	330	2 2	202	3 5	254 of labor cost
c) Lab expendables and	12	€	800	2	3600 - 1080 - 1080
miscellaneous Ambient air monitoring:					
a) Residential stations	4	EA	30,000	120	High-volume air samplers
b) Background database	,	<u>.</u>	20,000	02 2	;
	12	£	4,000	<b>4</b>	•

APPENUIX Y, EXHIBIT 1. (continued)

Comments	25% of labor cost	1		\$20/day x 27.5 d/mo 1 lb carbon/1000 gal for 17,000,000 gal; decon, quench,	runott 8 hr/d for 330 days	10% of facility capital cost	10 gpm x 60 min/hr x 24 hr/d	x 330 d; decon and ancil facility 130 kM x 24 h/d x 27.5 d/mo x \$0.048/kWn		Factor applied to subtotal Factor applied to subtotal and	contingencies Factor applied to subtotal + contingencies and GA			Transport from Borger, IX to	cuitport, 43 and back modules @ 72,000 lb or 36 tons	eacn
Cost (\$1000)	117	10		34	67 20	30	4	38		277 216	150 2,030			162	122	15
Unit Price	4,888 88	800		550 2.00	25.5	30,000	0.80	3,168		: :	1	1		000*9	125	15,000
Units	MO V	C-1		MO 1bs	HR 1000	rs S	1000	. C		::	;	RING COST	0STS	TRL	TON	TS
Quantity	24 330	12		12,000	2,640 17,000	-	4,752	12	T SUBTOTAL	: :	:	AND MONITO	P/REMOVAL C	27	972	-
ltem	Sampling collection:  a) Sampling technicians (2)  b) Per diem and general	expense c) Expendables and miscellaneous	FACILITY OPERATIONS	Water treatment operation: a) General b) Carbon replacement	<ul><li>c) Facility operator</li><li>d) Discharge to POIW</li></ul>	e) Maintenance (misc)	Water user charge	Electricity	COMMON OPL TING AND MONITORING COST SUBTOTAL	Contingencies (20%) General Administration (13%)	Contractor fee (8%)	CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST	CATEGORY 3: AER FACILITY SITE SETUP/REMOVAL COSTS	Transportation trailers to and	rrom site Erect AER and pretmt building	Startup/shakedown

APPENDIX Y, EXHIBIT 1. (continued)

	ltem	Quantity	Units	Unit Price	Cost (\$1000)	Соптепts
Rеmov	Remove AER and pretmt building	972	10N	100	91 396	27 modules @ 72,000 lb or 36 tons each
FAC IL	FACILITY SITE SETUP/REMOVAL COST SUBTOTAL	TOTAL				
Conti Gener	Contingencies (25%) General administration (13%)	: :	: :	::	99	Factor applied to subtotal Factor applied to subtotal and
Coatr	Contractor fee (8%)	;	1	;	604	contingencies Factor applied to subtotal and contingencies + GA
CATEG	1	MOVAL COSTS		,		
2	CAICGURT 4: AER FACILITI OPERALING AND MAINIENANCE COSTS	AND MAINIEN	ANCE COS	ا2		
Equip	EQUIPMENT USE CHARGE	14,500	QW QW	111,000	019.1	Pretreatment and AER equipment includes transportation time
OPERA	OPERATION OF AER FACILITY					
Ut 411						
8 Z	a) AER power requirements b) Ancillary power requirements	20,000 12	TON	52.80 6,336.00	1,056	1100 kWh/ton * \$.048/kWh 200 kWh * 24 h/d * 27.5 d/mo
(°)	Nitrogen	000,036	8	0.40	380	* p.048/kWh 200 scfm use rate during opera-
Đ	Water user charge	5,227	200	0.80	4	tion or ~4/50 ft/ton ligpm for ash quenching * 60 min/h
e)	e) Natural gas	12 23,760	1000 fr3	9.00	119	* 24 n/d * 330 d/y 3000 cf/h use rate during opera- tion or ~1200 ft3/ton
Main	Maintenance of AER:					
. a	Electrode replacement	20,000	FON	1,25	\$2	Replace @ 20,000 tons/electrone
57	Misc materials	20,000	T ON	23.00	25.	Replace & 2,000 tons/core
<del>9</del>		20,000	NO.	2.00	9	sh of equipment Capital Cost

APPENUIX Y, EXHIBIT 1. (continued)

	Item	Guantity	Units	Unit Price	(\$1000)	Comments
(a)	at AER facility Operations manager (1)	12	Q C	8,532.48	102	6 \$48,000/y x
च्या इंट	Maintenance personnel (2) Reactor operator (4)	24 24 48	2 <b>2</b> 2	4,614.29	111	ള വര
a Ç	Yard operator (4) Pretreatment area operator (4)	444	<b>2</b> 5	4,204.02	205 208 208	Salary @ \$23,650/y x burden Salary @ \$24,420/y x burden
g) h)	Relicf operator (2) Engineers/safety personnel	2 <b>4</b> 36	₩ ₩ ₩	<b>4,340.90 6,754.88</b>	104 243	Salary @ \$24,420/y x burden Salary @ \$38,000/y x burden
<del>-</del>	Per diem and general expense	330	0.r	923.48	305	25% of total labor cost for 21 employees
Health a) b	Physicals (2 per w Protective equip. Level C (20)	42 6,600	E E	600.00	25 417	
AER FAI	Level D (1)  AER FACILITY OPERATING AND MAINTENANCE COST SUBTOTAL	330 CE COST SUB	TO TAL	<b>69.9</b> 2	5,90 <u>5</u>	Furchase and disposal of protective equipment
Cont in Genera	Contingencies (20%) General administration (13%)	: :	: :	::	1,181	Factor applied to subtotal +
Contra	Contractor fee (8%)	ł	:	}	641 8,648	Factor applied to subtotal + contingencies and GA
CATEGOI TOTAL E	CATEGORY 4: TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS TOTAL ESTIMATED COST	ATING AND P	TAINTENAN	ICE COSTS	14,333	

APPENDIX Y, EXHIBIT 2. DETAILED COST ESTIMATE FOR 11,200 TON INTERIM SOIL STURAGE FACILITY

l tem	Quantity	Cost Units	Unit Price	(\$1000)	Comments
COMMON REMEDIAL COSTS					
GENERAL					
Mobilization and demobilization	- 0	L S	5 <b>%</b>	80	: :
Utilities connection Water supply	. – –		5,000.00	. ഗ ഗ	; ;
SITE PREPARATION					Storage for bulk wastepile of 11,260 tons of soil
Clear, strip and grub Grading and gravel surfacing	3,000	SY	1.50	5	1 1
Ditching for site runon Access roads	800 350	SY	0.56 5.70	0.5 2	20 ft wide with distance of 150 ft to AER facility
CONTAINMENT BASE					Double liner, leachate collection, leak-detection sys 160 ft x 160 ft
Gentertile	2.844	λ	1.85	S	
45 Mi. Hypalon liner	2,644	λŠ	6.75	19	1 1
Geotextile	2,844	ΣX	1.85	S	• •
12-inch leak detection gravel	948	ر ک	10.95	2 ٔ	
4-inch perforated PVC piping	622	k	20.60 20.00	<b>o</b> ~	; ;
8-inch reinforced concrete	535 <b>63</b> 2	ر د	150.00	95,	:
Impervious layer	2,844	SY	11.70	33	;
	2,844	λŠ	1.85	ro è	
	948	5	26.03	20	•
4-inch perforated PCV piping	333	<u>-</u>	20.00	~	
Geotextile	2,844	i ≿,	1.85	. vs	:

APPENUIX Y, EXHIBIT 2. (continued)

CONTROL DEVINERA SECOND DESCRIP RESERVATIONS DESCRIPTIONS DESCRIPTIONS

ltem	Quantity	Cost Units	Unit Price	(00013)	Comments
CONTAINMENT SIDEWALLS					5 ft nigh concrete wall around perimeter of wastepile
Impervious layer 10-inch reinforced concrete wall Vertical drain 4-inch perforated PVC pipe Waterproofing panels CONTAINMENT ENCLUSURE	400 100 400 640 400	SY SY SY	11.70 400.00 6.00 14.00 6.00	2	Around perimeter of wall
Pre-engineered steel building	25,600	SF	18.00	461	Steel frame building, assumed eave height 30 ft
HIGHTING AND ELECTRICAL HIVAC Double air-lock doors	00 <b>°c</b> 2	r 21 23	40,000.00 60,000.00	40 60	:::
BUILDING CLEANING AND RESTORATION					Decontaminate and reuse building instead of demolition
12-inch drainage gravel disposal	948	ζ	350.00	332	Dispose of gravel at pretreatment
Impervious layer disposal Decontaminate building	284 <b>4</b> 25 <b>,</b> 600	SY SF	36.00	102 154	
OPERATION AND MAINTENANCE					
Utilities Soil rehandling	11,200	0¥ 2 C.¥ 3	1,000.00	112	1 1

APPENDIX v, EXHIBIT 2. (concluded)

	Unit Price (\$1000) Comments	1,672.5		- 271.8 Factor applied to subtotal a		$\frac{194.0}{2.619.3}$ contingencies and GA
Cost	Units Unit		:	; ;	;	
	Quantity U		i	!	;	
	Item	CONSTRUCTION SUBTOTAL	Contingencies (25%)	General administration (13%)	Contractor fee (8%)	TOTAL INTERIM STORAGE FACILITY COST

APPENDIX Y, EXHIBIT 3. DETAILED COST ESTIMATE FOR TOLE AER OPERATIONS FOR 6 MONTHS

Comments			111			Salary @ \$4' 000/y x burden	Salary @ \$25,958/y x burden	Salary & \$25,169/y x Durden 25% of total labor gost for	4 employees	50 ft x 10 ft trailer		:	Personnel decon station	Lighting and heating	595	Factor applied to subtotal Factor applied to subtotal +	contingencies Factor applied to subtotal +	contingencies and GA
(\$1000)			15 10 666			51	55	38		2	נט	2	15	9	188	176	96	1,290
Unit Price			15,000.00	•		6,532.48	4,614.29	201.52		379.50	379.50	270.00	1,000.00	1,000.00				
Cost			ನನಕ			오	<u></u>	26		OW.	£	£	Q W	Q.				
Quantity	00515		9			9	<u>2</u>	165		9	9	9	12	9				হা
Item	AER FACILITY OPERATING AND MAINTENANCE COSTS	UE NE KAL	Second startup & shakedown AER equipment and facility decon Equipment use charge	STANDBY OLM COSTS	Labor at AER facility	Operations manager (1)	Maintenance personnel (2)	Per diem and general expense	Rental equipment remaining onsite	Office trailer	Employee trailer - breakroom	Excavation admin trailer	Decor. trailer rental (2)	Utilities	OPERATING AND MAINTENANCE COST SUBTOTAL	Contingencies (20%) General administration (13%)	Contractor fee (8%)	TOTAL COST DURING 6-MONTH IDLE OPERATIONS

APPENDIX Y, EXMIBIT 4. DETAILED COST ESTIMATE FOR AER TREATMENT OF 40,000 TONS OF SOIL

Cost Quantity Units Unit Price (\$1300) Comments			on and 1 LS 130,000 130 5% of Category 1 subtotal cost	s 1 LS 150,000 150 s		and equipment: inf facility 1 LS 300,000 300	23 M0 380 9	iler – breakroom 23 MO 360 9 50 ft x 10 ft trailer	23 MO 1,58i		89 000,89 51 1	5 EA	1 LS 20,000 20	1 LS 30,000 30	rvice allowance 1 LS 10,000 10		ion trailer 46 MO 1,000 46	of non 1 15 20 non 20
l ten	CATESORY 1: COMMON REMEDIAL COSTS	MUBILLIA	General mobilization and demobilization	Trial burn Community relations	CONSTRUCTION	Ancillary buildings and equipment: a) Water treatment facility	b) Office trailer	c). Employee trailer - breakroom	d) Forklift for material preparation	Utility upgrade:	a) Electrical services	b) Outdoor lights	_	d) Sewer Connection	f) Telephone service allowance	Excavation site buildings:	a) Uecontamination trailer Rental (2)	b) Vehicle decontamination

APPENDIX Y, EXMIBIT 4. (continued)

Comments		 3 for daily operation 12 yd <sup>3</sup> net volume, total storage for 7 days of operation 12 yd <sup>3</sup> net volume, total storage for 7 days of holding	8 hr/dcy for 420 days 8 hr/day for 420 days 8 hr/day for 420 days for each driver 8 hr/day for 420 days	8 hr/day for 420 days each worker 25% of H&S personnel cost 	Purchase and disposal of protective equipment Purchase and disposal of protective equipment for truck drivers
Cost (\$1000)		163 69 91 15 140	87 87 174 91	168 42 10 9	159 22 46
Unit Price		7,100 3,000 1,984 5,000 5,000	26 26 26 27	25 100 600 1,100	63 26 1,980
Units		MWW MWW FA EA	<b>.                                    </b>	EA EA	OF OF
Quantity		23 46 3 28 28 28	3,360 3,360 6,720 3,360	6,720 420 16 8	2,520 840 23
Item	EXCAVATION LOAD AND HAUL	Equipment:  a) Backhoe rental b) Tractor (with hoe ram) rental c) Rolloff truck rental (2) d) Rolloff boxes for operations e) Rolloff boxes for untreated Soil storage f) Rolloff boxes for treated soil storage	labor: a) Backhoe operator b) Tractor operator c) Rolloff truck driver (2) d) Excavation foreman	HEALTH AND SAFETY Health and safety personnel (2) Per diem and general expenses of H&S personnel Physicals (2/worker) Iraining	Level C Level D Dust control

APPENDIX & EXHIBIT 4. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
SITE RESTORATION					
Spreading treated soil:			,	;	
a) Dump truck	23	<b>£</b>	1,984	46	:
b) Iruck driver	3,360	至 ;	26	83	8 hr/day for 420 days
Regrade Site	84,700	<u>ک</u> د	æ.o.:	89	880 ft x 220 ft
lopsali placement Clean fill	3,585 2,055	ح د	7.00	4 <del>4</del>	6-inch depth x 880 ft x 220 ft 15% of 37,600 vd <sup>3</sup> less top sail
	•				placement
Erosion matting Spacing and receptation	84,700	S.	0.80	99 110	1 *
Salvage of rolloff boxes	65 29	EA.	2,500	- 148	50% of cost, 3 operations, 28
COMMON REMEDIAL COST SUBTOTAL				2,731	untreated soil, and 28 treated soil
Contingencies (25%)	:	;	;	683	Factor applied to subtotal
General administration (13%)	;	;	;	444	Factor applied to subtotal +
Contractor fee (8%)	;	:	:	309	contingencies factor applied to subtotal +
CATEGORY :: TOTAL COMMON REMEDIAL CUST	1503			4,167	CONTINGENCIES and GA
CATEGORY 2: COMMON OPERATING AND MONITORING COSTS	ONITORING CO	STS			
GENERAL					
On-scene coordinator Per diem and general expense	23 640	O.Y.	5,500 50	127	\$25/hr x 8 hr/Gay x 27.5 day/mo. 25% of labor cost
MONITORING AND ANALYSIS PROGRAM					
Onsite lab facility	<b>,</b>	rs T	250,000	250	Includes enclosure, high resolution GCIMS, ancillary
Analytical Lab Operation: a) Chemists (4) b) Per diem and general expense c) Lab expendablas and miscellaneous	92 640 23	M V W	8,333 300 800	767 192 18	equipment 25% of labor cost

APPENDIX Y, EXHIBIT 4. (continued)

lter	Quantity Units	Units	Unit Price	Cost (\$1000)	Comments
Ambient sir monitoring: a) Residential stations b) Background database c) Air sampling snalysis	23	EA LS MO	30,000 20,000 4,000	120 20 92	High volume air samplers 
0 0 79	46 640	μ Ωλ	4, 888 63	225	25% of !abor cost
c) cxpendables and miscellaneous FACILITY OPERATIONS	57	S.	308	<u>~</u>	:
Water Treatment Operation: a) General b) Carbon replacement	31,000	MO 1bs	550	13	\$20/day x 27.5 day/mo. 1 lb carbon/1,050 gal, 31,000,000 gal; decon, quench,
c) Facility operator d) Discharge to POIW	5,064 31,000	1000 1000	25.5	129	runoff 8 hr/day for <b>633 day</b> s
e) Maintenance (misc)	-	Į.s.	30,000	30	10% of facility capital cost
Water User Charge	9,115	KGAL	0.80	_	10 gpm x 60 min/hr x 24 hr/day x 633 days; decon and ancil
Electricity	23	2	3,168	73	Tacility 100 kW × 24 h/d × 27.5 d/mo.
COMMON OPERATING AND MONITORING COST SUBTOTAL	SUBTOTAL			2,268	x ≯0.048/kmn
Contingencies (20%) General administration (13%)	::		;;	454	Factor applied to subtotal
Contractor fee (8%)	;	;	ŧ	246	contingencies Factor applied to subtotal + contingencies and GA
CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST	ND MONITORI	NG COST		3,322	

APPENDIX Y, EXHIBIT 4. (continue)

Connents		Transport from Borger, 14 to	Gulfport, MS and back 27 medules @ 72,000 lb or 36 tons	each 27 modules @ 72,000 lb or 36 tens	each	Factor applied to subtotal Factor applied to subtotal +	contingencies Factur applied to subtotal + contingerries and GA			Pretreatment and AER equipment including transportation fine		1100 kwh/ton x \$.048/kwh 200 kwh x 241/d x 27.54/ma	x .048/kWh 200 scfm use_rate during eperation	or 4750 ft3/ton 11 gpm for ash quenching	$\times$ 60 min/h $\times$ 24 h/d $\times$ 633 days 3000 cf/h use rate during operation or 1200 ft $^3$ /tor
Cost (\$1000)		162	122	15 97	396	99	45	604		2,664		2,112	760	κο	240
Unit Price		6,000	125	15,000 100		1 :	1		STS	111,000.00		52.80 6,336.00	0.40	08.0	5.00
Units	315	TRL	10N	LS		::	;		ANCE CO	о <del>м</del>		TON MO	001	1000	1000 ft <sup>3</sup>
Quantity Units	P/RE-NUVAL CO	27	912	972	STUTAL	; ;	;	MUVAL COSTS	AND MAINTEN	24		40,000	1,300,000	10,027 1000	48,000
Item	CATEGORY 3: AER FACILITY SITE SETUP/RE BOVAL COSTS	iransportation trailers to and from site	Erect AER and pretmt building	Startup/shakedown Remove AER and pretmt building	FACILITY SITE SETUP/REHOVAL COST SUSTOTAL	Contingencies (25%) General administration (13%)	Contractor fee (8%)	CATEGORY 3: TOTAL AER SITE SETUP/REMOVAL COSTS	CATEGORY 4: AER FACILITY OPERATING AND MAINTENANCE COSTS	EQUIPMENT USE CHARGE	OPERATION OF AER FACILITY	Utilities: AER power requirements Ancillary power requirements	Nitrogen	Water user charge	Natural gas

APPENDIX Y EXHIBIT 4. (concluded)

İtem	Quantity	Units	Unit Price	Cost (\$1000)	Comments
Maintenance of AER: Electrode replacement Core replacement Miscellaneous materials Baghouse and carbon replacement	40,000 40,000 40,000	TON TON TON	1.25 25.30 6.00 2.00	50 1,000 240 80	Replace @ 20,000 tons/electrode Replace @ 2,000 tons/core 3% of equipment capital cost
Labor at AER facility: Operations manager (1) Secretary (1) Maintenance personnel (2) Reactor operator (4) Yard operator (4)	23 46 92 92	M M M M M M M M M M M M M M M M M M M	8,532.48 2,844.16 4,614.29 4,477.60 4,204.02	196 65 212 412 387	6 \$16000/y x to \$16000/y x to \$25958/y x to \$25189/y x to \$25189/y x to \$23650/y x to
Pretreatment area operator (4) Relief operator (2) Engineers/safety personnel (3) Per diem and general expense	92 46 69 633	04 0 0 V	4,340.90 4,340.90 6,754.88 924.02	399 200 466 584	Salary @ \$24420/y x burden Salary @ \$24420/y x burden Salary @ \$38000/y x burden 25% of total labor cost for 21 employees
Health and safety program: Physicals (2 per worker) Protective equip Level C (20) Protective equip Level 0 (1)	42 12,650 633	EA MD MD	6v0.00 63.25 26.45	25 800 17	Purchase and disposal of protective equipment Purchase and disposal of protective equipment
AER FACILITY OPERATING AND MAINTENANCE SUBTOTAL CUST	CE SUBTOTAL	COST		11,063	
Contingencies (20%) General administration (13%)	::	::	: :	2,213	Factor applied to subtotal Factor applied to subtotal and continuencies
Contractor fee (8%)  TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS TOTAL ESTIMATED COST	 NTENANCE CO	 SIS	:	1,200 16,202 24,295	Factor applied to subt <b>otal a</b> nd contingencies and GA

APPENDIX Y, EXHIBIT 5. UETAILED COST ESTIMATE FUR MER TREATMENT OF 10,000 TOHS OF SOIL

SCASS TOSOCCAPTS NAME AND SASTED A COLCANDENCE OF THE PROSESS AND AND SECOND SASTED SASTED SASTED OF THE SASTED OF THE SASTED OF THE SASTED SA

APPENDIX Y, EXHIBIT 5. (continued)

Comments	3 for daily operation 12 yd <sup>3</sup> net vol, total storage for 7 days of holding	8 hr/day for 130 days 8 hr/day for 130 days 8 hr/day for 130 days for each driver 8 hr/day for 130 days	8 hr/day for 130 days each worker 25% of H&S personnel cost 	Purchase and disposal of protective equipment Purchase and disposal of protective equipment for truck drivers
Cost (\$1000)	50 21 28 15 140 140	27 27 54 28	52 13 10	49
Unit Price	7,100 3,000 1,984 5,000 5,000	26 26 26 27	25 100 600 1,100	63 26 1,980
Units	M M M E E A E A	# # # #	HR OY EA	M M OM
Quantity	7 7 14 13 28 28	1,040 1,040 2,080 1,040	2,080 130 16	260
Item EXCAVATION LOAD AND HAUL	Equipment:  a) Backhoe rental b) Tractor (with hoe ram) rental c) Rolloff truck rental (2) d) Rolloff boxes for operations e) Rolloff boxes for untreated soil storage f) Rolloff boxes for treated soil storage	Labor: a) Backhoe operator b) Tractor operator c) Rolloff truck drivers (2) d) Excavation foreman	Health and safety personnel (2) Per diem and general expense for H&S personnel Physicals (2/worker)	Protective Equipment Level C (6 sets) Level D (2 sets) Dust Control

APPENDIX Y, EXHIBIT 5. (continued)

Comments	;	8 hr/day for 130 days 880 ft x 220 ft	15% of 9,400 yd3	1 1	50% of cost, 3 operations, 28 untreated soil, and 28 treated	soil	Factor applied to subtotal	Factor applied to subtotal +	Contingencies Factor applied to subtotal +	contingencies and bA			\$25/hr x 8 hr/way x 27.5 day/mo. 25% of labor cost		Includes enclosure, high resolution GC/MS, ancillary		25% of labor cost	:
(00013)	4.	27	<u>6</u>	89 2	148	1,649	412	268	186	2,515			39 10		250		233 58	ယ
Unit Price	489.	26	13.50	0.80	- 2,500		;	;	:				5,500 50		250,000		8,333 303	800
Cost Units	7 <del>0</del>	¥ (s	<u>ن</u> د	ر د در	EA .		. :	:	:		STS		M0		S.		D¥ D¥	MO
Quantity	1	1,040 84,700	1,410	84,700	65		;	:	;	2051	ONITORING CO		192		<del>-</del>		28 192	1
Item	Spreading treated soil: a) Dump truck	b) fruck driver Regrade site	Topsoil replacement	Erosion matting Seeding and revenetation	Salvage of rolloff boxes	COMMON REMEDIAL COST SUBTOTAL	Contingencies (25%)	General administration (13%)	Contractor fee (8%)	CATEGORY 1: TOTAL COMMON REMEDIAL COST	CATEGORY 2: COMON OPERATING AND MONITORING COSTS	GENERAL	On-scene coordinator Per diem and general expense	MONITORING AND ANALYSIS PROGRAM	Onsite lab facility	Analytical Lab Operation:	<ul> <li>a) Chemists (4)</li> <li>b) Per diem and general Pannole</li> </ul>	c) Lab expendables and miscellaneous

APPENDIX Y, EXHIBIT 5. (continued)

				•			>.				
Comments	High-volume air samplers 	25% of labor cost		\$20/day x 27.5 day/mo. 1 1b carbon/1000 gal, 10,000,000 gal; decon, quench,	runoti 8 hr/day for 193 days	10% of facility capital cost	10 gpm x 60 min/hr x 24 hr/day x 193 days; decon and ancil	100 kW x 24 h/3 x 27.5 d/mo. x \$0.048/kWh		Factor applied to subtotal +	Factor applied to subtotaland contingencies and GA
Cost (\$1000)	120 20 28	68 17 6		20	39	30	2	22	984	197	107
Unit Price	30,000 20,000 4,000	4,888 888 800		550 2.00	25.5 1.20	30,000	0.80	3,168		;;	<u> </u>
Units	EA LS MO	M NV MO		M0 16s	HR 1000	ga i	1000 gal	<b>₩</b>		: :	 11NG COST
Quantity Units	4	14 192 7		10,000	1,544	-	2,779	7	SUBTOTAL	::	AND MONITOR
Item	Ambient air monitoring:  a) Residential stations  b) Background database  c) Air sampling analysis	Sampling Collection:  a) Sampling technicians (2)  b) Per diem and general expense  c) Expendables and miscellaneous	FACILITY OPERATIONS	Water treatment operation: a) General b) Carbon replacement	c) Facility operator d) Discharge to POTW	e) Maintenance (misc)	Water User Charge	Electricity	COMMON DEFRATING AND MONITORING COST SUBTOTAL	Contingencies (20%) General administration (13%)	Contractor fee (8%)  CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST

APPENDIX (, EXHIBIT 5. (continued)

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	ltem	Quantity Units	Units	Unit Price	Cost (\$1000)	Comments
CATE	CATEGORY 3: AER FACILITY SITE SETUP/REMOVAL COSTS	/REMOVAL CO	STS			
Tran	Transportation trailers to and	27	<b>1</b> RL	000 <b>.9</b>	162	Transport from Borger, IX to
Erec	from Site Erect AER and pretmt building	972	10M	125	122	27 modules @ 72,000 lb or 36 tons
Star	Startup/shakedown Remove AER and pretmt building	972	LS TON	15,000	15 97	each 27 modules @ 72,000 lb or 36 tons
FACI	FACILITY SITE SETUP/REMOVAL COST SUBTOTAL	TOTAL			396	each
Cont	Contingencies (25%) General administration (13%)	: :	: :	: :	97 64	Factor applied to subtotal
Conti	Contractor fee (8%)	i	:	ţ	45	Factor applied to subtotal and
CATE	CATEGORY 3: TOTAL AER SITE SETUP/REMOVAL COSTS	MOVAL COSTS			604	contingencies and ba
CATE	CATEGORY 4: AER FACILITY OPERATING AND MAINTENANCE COSTS	AND MAINTEN	ANCE CO	SIS		
EQUI	EQUIPMENT USE CHARGE	∞	£	111,000.53	808	Pretreatment and AER equipment, includes transportation time
OP ER,	OPERATION OF AER FACILITY					
Ut il	Utilities: a) AER power requirements b) Ancillary power requirements	10,000	10M	52.80 <b>6,</b> 336.00	528 44	1100 kWh/ton x \$.048/kWh 200 kWh x 24 h/d x 27.5d/mo
(c)	Nitrogen	475,000	25.	0.40	190	200 sofm use rate during operation
P	d) Water user charge	3,057	200	0.80	2	li gpm for telicon
<b>e</b>	e) Natural gas	12,000	1000	5.00	70	$x$ of min/n $x \le 4$ n/d $x$ 193 days 30 $^{(4)}$ cf/h use rate during operation or 1200 ft $^3$ /t-n

APPERUIX Y, EXHIBIT 5. (continued)

SOSSI ESSECTED CLUCKEN GERROOF DORRECHEN SON MINISTERIAL KROOF WAS SELECTED SON MINISTERIAL

Comments	Replace @ 20,000 tons/electrode Replace @ 2,000 tons/core 3% of equipment capital cost	Salary @ \$48000/y x 202% overhead	8 3.0% wan Salary @ \$16000/y x 202% overhead	x 5.6% toky Salary @ 125958/y x 202% overhead	Salay # 25189/y x 202% overhead	x 3.0% 36.4 Salary @ \$23650/y x 202% overhead	Salary @ \$24420/y x 202% overhead	x 5.6% 66A Salary @ \$ 1420/y x 202% overhead	Sa 2.0% back Sa 2.0% 38000/y x 202% overhead	A 3.0% bak SLX of total labor cost for 21 employres	Purchase and disposal of	protective equipment Purchase and disposal of protective equipment
Cost (\$1000)	13 250 60 20	09	20	99	125	118	122	19	142	178	25 244	35
Unit Price	1.25 25.00 6.00 2.00	8,532.43	2,844.16	4,614.29	4,477.60	4,204.02	4,340.90	4,340.90	6,754.88	925.97	600.00 63.25	26.45
Units	10N 10N 10N 10N	NO	₩ O	MO	MO	ON ON	œ.	æ	<b>W</b> 0	ργ	EA	Q.
Quant ity	10,000 10,000 10,000	7	1	14	28	28	28	14	21	193	42 3,860	193
Iten	Maintenance of AER:  a) Electrode replacement b) Core replacement c) Miscellaneous materials d) Saghouse and carbon replacement	Labor at AER facility: a) Operations manager (1)	b) Secretary (1)	c) Maintenance personnel (2)	d) Reactor operator (4)	e) Yard operator (4)	f) Pretreatment area operator (4)	g) Relief operator (2)	h) Engineers/safety personnel (3)	i) Per diem and general expense	Health and safety program:  a) Physicals (2 per worker)  b) Protective equip Level C	c) Protective equip Level D (1)

APPENDIX Y, E\*HIBIT 5. (concluded)

t 00) Comments	30	646 Factor applied to subtotal		contingencies 350 Factor applied to subtotal + contingencies and GA	3,	16
Cost (\$1000)	3,230	9	2	er e	4,730	9,291
Unit Price		;	;	;	ANCE COSTS	
Units	1800	:	;	;	AINTENA	
Item Quantity Units	AER FACILITY OPERATING AND MAINTENANCE SUBTOTAL CUST	Contingencies (20%)	General administration (13%)	Contractor fee (8%)	CATEGORY 4: TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS	TOTAL ESTIMATED COST

# APPENDIX Z

UPDATED COST ESTIMATE FOR THE J. M. HUBER METHOD OF PROCESSING TOXIC WASTE AT THE GULFPORT, MISSISSIPPI, SITE



# INTEROFFICE CORRESPONDENCE

Date:

March 20, 1987

To

R. W. Thomas

From:

H. J. Welland 110

Subject:

UPDATED COST ESTIMATE FOR THE J. M. HUBER METHOD OF PROCESSING TOXIC

WASTE AT THE GULFPORT, MISSISSIPPI SITE - HJW-13-87

Cost Estimating has updated the cost estimate for the J. M. Huber Corp. Category 1, Common Remedial Costs and Category 2, Common Operating and Maintenance Costs for processing the toxic waste located at the Gulfport, Mississippi site. A copy of the assumptions stated in the original transmittal letter (K. A. !enjum to R. W. Thomas letter dated June 18, 1986) is included for information. The changes considered in this update are:

- 1. The size of the excavation area and the associated regrading costs were revised to agree with the latest contaminated volume and depth estimates.
- 2. Per diem expenses have been added for the Chemists and the On Scene Coordinator because it was felt it would be more efficient if trained personnel were brought to the job site rather than trying to find or train local personnel.
- 3. The full time decontamination worker has been deleted because only minor, sporadic decontamination will be required during the excavation, which the equipment operator can perform. The major decontamination effort will occur at the conclusion of the excavation effort. The original estimate assumed that the excavation personnel would participate in the final decontamination effort, thus full-time decontamination personnel are not required.
- The costs for general mobilization have been revised to 95 percent of the Category 1 subtotal costs.
- 5. A contractor fee of 8 per cent has been added.

The detailed backup sheets have been revised and are attached to this letter.

hjw

Attachments: As Stated

cc: Central Files
Project File #8076-A/

353

"Providing research and development services to the government"

Assumptions taken from letter from k. A. Henjum to R. W. Thomas, dated June 18, 1986:

- 1. Facility/equipment site development can take place as a normal construction operation. Since the facility/equipment site is in a clean area, there is no dange, of contamination spread to workers as long as dust-creating active ies are not occurring on the excavation site. Unit prices for equipment area and yard floor slabs need not reflect added labor costs due to personal-protective clothing effects on workers, and thus were lowered.
- 2. Excavation site preparation was eliminated. The excavation site has been stabilized with soil cement and additional fill material. Grasses have grown through the surface of the site. It is assumed that the ground is capable of supporting heavy equipment, thus eliminating the need for on-site roads. Contaminated soil on the surface of the site cannot be removed by clearing and grubbing as is the normal method of removing grasses from an excavation site because the removed soil would constitute a hazardous waste. It is assumed that the soil pretreatment facility can handle the amount of grass excavated from the site. The excavation site is very flat with ditches on site to control water flow on the site. These ditches are monitored for contamination. Further ditching for site run on will be unnecessary.
- 3. Soil stabilization occurs to a depth of six to twelve inches on site. Contamination in this soil is expected to reach a maximum depth of approximately 1 1/2 feet. This soil will need to be broken up before it can be excavated. A tractor, such as a Case 580 with a hoe ram attachment, is suggested for this work. The hoe ram could break up sufficient soil for one day's excavation, leaving the rest of the site intact. In this way, the soil stabilization can be taken advantage of to minimize the spread of contaminated dust from the excavation site while maintaining a surrace for hauling the excavated soil offsite.
- 4. There is one venicle decontamination station. This station will be capable of decontaminating the equipment which will be removed from the site at the end of the project.
- Excavation will be done five days a week in an eight-hour day. It is assumed that loading of soil will occur in a five-hour period. Assume that 220 days will be available to work, allowing for storms, holidays and equipment down time. Excavation equipment is oversized to allow for doubling the daily requirement of the treatment facility to allow the soil supply to catch up after periods of depletion caused by excavation down time.

- 6. Excavation crew rates were lowered to reflect DOE and Davis-Bacon labor rates for that area. Per diem and general expense allowance of 25 percent of labor costs was included in the labor hourly rates. The rate using local operating engineers and training them within the handling of hazardous waste is a minimum of 14 percent less costly than using a hazardous waste trained operating engineer and paying him per diem.
- 7. Mapping of the excavation site contamination has been performed to provide a preliminary scope for excavation. For the purpose of this estimate, it was assumed that the depth of the base case excavation area will double for twice the base soil volume or will be halved for one half of the base soil volume. This assumption is based on the expectation that subsurface contamination will not cover a larger area than the known surface contamination.
- 8. After treatment, the original soil will be returned to the excavation site. It is expected that fifteen percent of the original soil volume will be lost during treatment. Clean fill top soil of appropriate quality will be used to fill the remaining volume of the excavation site so that the site may be recontoured for its final intended use as a parking area.
- 9. The federal facility which has the waste site will provide several services. Specifically, these are:
  - o All permitting associated with the work
  - o All licensing and ash delisting procedures
  - o A site warehouse
  - A site parking area
  - A prepared area for placement of the treatment facility
- 10. The costs for bonding and insurance is included in the overhead costs.
- 11. The estimate does not include costs for footings, foundations or slabs to place the process equipment on. It is assumed that the stabilized soil is adequate for all intended loads.
- 12. One other area of discrepancy is that CH2M Hill maintains that the soil may not become completely delistable and thus a disposal site must be provided for the treated soil and fill material will be needed for the excavation site. This estimate was prepared on the assumption that the soil will be completely delistable and can be returned to the original excavation site or else nothing will be done and the soil will remain undisturbed in its present state.

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